ARKANSAS STATE CRIME LABORATORY

FORENSIC CHEMISTRY

QUALITY MANUAL

DIRECTOR:

KERMIT B. CHANNELL, II

CONTENTS

1 SCOPE

This manual follows the requirements specified by ANSI-ASQ National Accreditation Board (ANAB), which is based on the ISO/IEC 17025:2017 standards and the 2017 ANAB ISO/IEC 17025:2017 — Forensic Science Testing and Calibration Laboratories Accreditation Requirements (AR 3125).

The *Forensic Chemistry Quality Manual* is written specifically for the analysts working in the Drug Section and performing analyses in the following areas:

- Controlled Substance Analysis
- Clandestine Laboratory Analysis
- General Chemical Testing
- Tampering
- Quantitative Determination (THC only)

Evidence selection, for analysis, is based on the analyst's training and experience.

1.1 INTERNATIONAL STANDARD: GENERAL REQUIREMENTS

See *ASCL-DOC-01 Quality Manual*.

1.2 INTERNATIONAL STANDARD: SCOPE

See *ASCL-DOC-01 Quality Manual*.

1.2.1 ANAB PROGRAM

See *ASCL-DOC-01 Quality Manual*.

2 NORMATIVE REFERENCES

The Forensic Chemistry section follows applicable references listed in *ASCL-DOC-01 Quality Manual*.

Additional references include:

- *ASCL-DOC-01 Quality Manual*
- Arkansas Criminal Code for Controlled Substances
- Arkansas Controlled Substance List

3 TERMS AND DEFINITIONS

Additions to *ASCL-DOC-01 Quality Manual* are listed below.

CONTROLLED SUBSTANCE

A drug or chemical, whose manufacture, possession, or use is regulated by a government.

CUTTING AGENT (DILUENT, ADULTERANT)

A substance added to reduce the purity of another substance.

EVIDENCE

Items submitted to the laboratory for analysis.

4 GENERAL REQUIREMENTS

4.1 IMPARTALITY

See *ASCL-DOC-01 Quality Manual.*

4.2 CONFIDENTIALITY

Investigative information may not be released until after a technical review has been completed. Should information need to be released, before the case is completed and a technical review has been conducted, the following shall occur:

- Separate competent individual shall review data to confirm findings to be released (e.g., GCMS, FTIR, TLC, GCRT results)
- Reviewer of data shall document what was reviewed (this may be done by memo or via an email to the case analyst stating what data was reviewed and what may be released)
- During the release of information, the analyst shall communicate their results are preliminary and a final report shall be completed and released upon completion of analysis

5 STRUCTURAL REQUIREMENTS

5.1 ESTABLISHMENT

Act 517 of 1977 established the Arkansas State Crime Laboratory (ASCL) via A. C. A. § 12-12-301.

5.2 MANAGEMENT

The Arkansas State Crime Laboratory is managed by the Director, who has overall responsibility for the laboratory.

For 5.2.1 – 5.2.8 See *ASCL-DOC-01 Quality Manual*.

5.2.9 OTHER STAFF (FORENSIC CHEMISTRY STAFF)

5.2.9.1 CHIEF FORENSIC CHEMIST

QUALIFICATIONS

The position requires the formal education equivalent of a bachelor's degree in chemistry; plus five years of experience in a chemical laboratory, including two years in a forensic laboratory, or a related field. Other job related education and/or experience may be substituted for all or part of these basic requirements upon approval of the Assistant Director.

AUTHORITIES AND RESPONSIBILITIES

The Chief Forensic Chemist is under administrative direction and is responsible for the activities of the Forensic Drug Chemistry Section in Little Rock and satellite laboratories. The Chief Forensic Chemist has the overall responsibility for the technical operations and the provision of the resources needed to ensure the quality of the laboratory operations. The Chief Forensic Chemist will have the appropriate technical training and technical experience in the drug section.
The Chief Forensic Chemist will have regular contact with crime laboratory staff, frequent contact with law enforcement agencies and judicial officials, and limited contact with the public. The Chief Forensic Chemist ensures compliance with ANAB International requirements by implementing lab wide policies and overseeing the section's quality assurance program.

- Supervises a large-sized technical staff of Forensic Chemists including interviewing applicants and recommending for hire, approving leave, making work assignments, training employees and evaluating the performance of employees
- Assists with developing laboratory policies and procedures, develops short and long-range operational plans for the forensic chemistry section, monitors operational activities by conducting staff meetings to disseminate information and reviewing and approving reports and compiles and submits statistical reports

- Manages the controlled substances authorized by the Drug Enforcement Administration (DEA) to be used during the process for comparing pure samples of controlled substances with findings to establish standards and maintains a log of the controlled substances used during testing including dates, amounts, and the name of the chemist requisitioning the substance

- Performs qualitative and quantitative forensic chemical analysis of known and unknown substances received from law enforcement agencies to determine the content of the substances using standardized laboratory methods and instruments, and documents procedure and results
- Presents expert forensic testimony in court on the chemical analytical methodology used to analyze evidence and analysis results, supervises pretrial conferences, and provides consultation to law enforcement and judicial officials on evidence collection and preservation method
- Compiles and interprets data obtained from analytical instruments, reviews and approves scientific forensic reports of section chemists, and writes conclusive scientific forensic reports
- Provides classroom instruction for law enforcement officers at seminars and courses statewide on drug identification, collection of evidence, and clandestine drug laboratory investigations
- Conducts research studies and validates new forensic analytical procedures, reviews current scientific literature and attends and participates in meetings and seminars to keep abreast of new technologies and procedures in the field.
- Performs related responsibilities as required or assigned

5.2.9.2 TECHNICAL LEADER

The Technical Leader is in charge of and accountable for the quality and training for the Forensic Chemistry section. The Technical Leader serves in this role for both the Little Rock and Lowell locations and is assisted by the training officers at each location.

QUALIFICATIONS

The position requires the formal education equivalent of a bachelor's degree in chemistry; plus three years of experience in a chemical laboratory, including two years in a forensic laboratory, or a related field. Other job related education and/or experience may be substituted for all or part of these basic requirements upon approval of the Assistant Director. The technical lead is in charge of quality and training for the Forensic Chemistry Section.

NEW TECHNICAL LEADER APPOINTMENTS

Any Forensic Chemistry Technical Leader appointed on or after September 1, 2022 shall be a currently or previously qualified analyst in each technology utilized in the section, or have documented training in each technology utilized in the section within one year of appointment.

Newly appointed technical leaders shall be responsible for the following within one year of appointment:

1) Review of validation studies and analytical procedures currently used by the section
2) Review of educational and training records of currently qualified analysts and technical reviewers
3) ANAB assessor training (could be longer than 1 year depending on availability of training)

AUTHORITIES AND RESPONSIBILITES

- All duties of Forensic Chemist
- Ensures that instrument, balance, chemical/reagent and reference material logs are recorded appropriately; prepares and records proficiency tests; maintains reference material inventory
- Helps maintain and update the section's manuals and documents
- Monitors the section's practices for compliance with the section's SOP
- Ensures the validation and verification of new technical procedures
- Works with lab wide Quality Manager and Chief Forensic Chemist to seek ways to improve the quality system
- Oversees the training program and training officers
- Works with Chief Forensic Chemist to seek ways to improve the training program.
- Coordinates intern program projects

AUTHORIZATIONS

- Can recommend suspension and resumption of Forensic Chemistry technical operations for the laboratory or an individual
- Oversees the technical operations of the Forensic Chemistry section
- Approves method development, modification, verification, and/or validation

5.2.9.3 FORENSIC CHEMIST

QUALIFICATIONS

The Forensic Chemist must possess a baccalaureate or advanced degree in natural science or closely related field with knowledge of the principles and practices of chemistry, chemical analysis and laboratory equipment. Before performing casework, the forensic chemist will be required to successfully complete a training program that will include competency sample testing, written and oral examination, and a mock trial (this training program is waived for forensic chemists hired before the issuance of the quality program and can be modified, based on experience, for those hired after the issuance of the quality program). This position is governed by state and federal laws and agency policy.

AUTHORITIES AND RESPONSIBILITIES

- Process evidence suspected of containing controlled substance(s) submitted to the ASCL by law enforcement agencies
- Present expert forensic testimony in court on chemical analytical methodology used to analyze evidence and obtain results

- Participate in pretrial conferences and provide consultation to law enforcement and judicial officials on evidence collection, preservation methods and analysis results.
- Verify the correct operation of scientific instruments and perform routine maintenance as needed. Prepare and verify reference materials and reagents according to established guidelines
- Review current scientific literature. Study and validate new forensic analytical procedures and modify new and/or old procedures as necessary
- Attend and participate in professional meetings and seminars to keep abreast of new technologies and methods in chemistry
- Assist with training new laboratory staff in performing standardized laboratory test.
- Perform related responsibilities as required or assigned
- Process evidence suspected of containing controlled substance(s) or chemicals that are suspected of being used to manufacture a controlled substance submitted to the ASCL by law enforcement agencies (Illicit Lab chemist only)
- On call to aid law enforcement in safely dismantling illicit laboratories and collecting representative samples (Illicit Lab chemist only)
- Instruct law enforcement officials on proper methods of confiscating, preserving, and disposing of toxic chemicals, waste and equipment found in clandestine drug laboratories (Illicit Lab chemist only)

5.2.9.4 FORENSIC TECHNICIAN

QUALIFICATIONS

This position requires the formal education equivalent of a high school degree.

AUTHORITIES AND RESPONSIBILITIES

- Perform performance verifications on instrumentation
- Transport evidence between Secure Storage and FC Secure Storage
- Assess submission sheets
- Prepare chemicals, reagents, reference materials, and controls needed for casework
- Assign daily reviews to Forensic Chemistry personnel
- Conduct annual inventory of controlled reference materials
- Perform related responsibilities as required or assigned
- Sampling of bulk cases, when trained and authorized

5.2.9.5 HEALTH AND SAFETY OFFICER

- Conducts monthly safety inspections and ensuring that proper practices and procedures are being followed in the section
- Assists with safety duties assigned by lab wide Health and Safety Manager

- Works with the lab wide Health and Safety Manager to seek ways to improve the safety program

5.2.9.6 TRAINING OFFICER

- Makes sure reading material for new hires is relevant and up to date
- Helps maintain and update the training manual and forms
- Devises a training schedule for new hires
- Works with Technical Leader to generate ideas for continued training for the section and ways to improve the training program for new hires
- Makes competency samples for new hires

5.3 SCOPE OF LABORATORY ACTIVITIES

See *ASCL-DOC-01 Quality Manual*.

5.4 NORMATIVE DOCUMENTS

See *ASCL-DOC-01 Quality Manual*.

5.5 LABORATORY OPERATIONS

See *ASCL-DOC-01 Quality Manual*.

5.6 QUALITY MANAGEMENT

See *ASCL-DOC-01 Quality Manual*.

5.7 MANAGEMENT SYSTEM COMMUNICATION AND INTEGRITY

See *ASCL-DOC-01 Quality Manual*.

6 RESOURCES REQUIREMENTS

6.1 GENERAL

See *ASCL-DOC-01 Quality Manual*.

6.2 PERSONNEL

6.2.1 GENERAL

See *ASCL-DOC-01 Quality Manual*.

6.2.2 COMPETENCE REQUIREMENTS

See *ASCL-DOC-01 Quality Manual*.

6.2.2.1 ANALYST/EXAMINER EDUCATIONAL REQUIREMENTS

See *ASCL-DOC-01 Quality Manual*.

6.2.2.2 TRAINING PROGRAM

The Forensic Chemistry Training Program is normally completed over a minimum of fourteen weeks. For analysts with prior experience, the training may be truncated with the approval of the supervisor and the Assistant Director.

See *ASCL-DOC-01 Quality Manual*, *DRG-DOC-02 Training Manual,* and *ASCL-DOC-03 ASCL New Analyst/Technician Training Manual*.

The Forensic Chemistry Section encourages the distribution and review of current literature. To this end, a literature folder is provided on the shared Drug network drive, to which literature is periodically added. Additionally, new literature may be distributed by email.

6.2.3 COMPETENCE OF STAFF

See *ASCL-DOC-01 Quality Manual* and *DRG-DOC-02 Training Manual*.

6.2.3.1 COMPETENCY TESTING

See *ASCL-DOC-01 Quality Manual* and *DRG-DOC-02 Training Manual*.

6.2.3.2 COMPETENCY–TESTED ACTIVITES

See *ASCL-DOC-01 Quality Manual* and *DRG-DOC-02 Training Manual*.

6.2.4 DUTIES, RESPONSIBILITIES, AND AUTHORITIES

See job descriptions in section 5.2 of this manual.

6.2.5 PERSONNEL REQUIREMENTS

See *ASCL-DOC-01 Quality Manual*.

6.2.6 AUTHORIZATIONS

See *ASCL-DOC-01 Quality Manual*.

6.3 FACILITIES AND ENVIRONMENTAL CONDITIONS

6.3.1 GENERAL

See *ASCL-DOC-01 Quality Manual*.

6.3.2 DOCUMENTATION

See *ASCL-DOC-01 Quality Manual*.

6.3.3 MONITORING RECORDS

A record of the temperature conditions for all Certified Reference Material storage locations within the section will be maintained (*DRUG-FORM-47 Temperature Log)*. Storage location conditions should be below 0°C. If storage conditions deviate from that specification for an extended time period, the cause will be assessed and any necessary action will be taken. Thermometers are assessed and replaced as needed.

6.3.4 CONTROL OF FACILITIES

See *ASCL-DOC-01 Quality Manual*.

6.3.4.1 ACCESS

LITTLE ROCK

The Forensic Chemistry section has a Secure Storage room within the section for storage of evidence prior to case assignment. This area is accessible by key; those keys are assigned to Forensic Chemists, Forensic Chemistry Supervisors/Technical Lead, and the Forensic Chemistry Technician.

Each Forensic Chemist has lockable areas to store assigned casework evidence. The Chief Forensic Chemist has access to these storage areas.

The Forensic Chemistry section has a key box containing cabinet keys and sections door keys. The key to the section key box is kept by the Chief Forensic Chemist. A log must be kept when keys are added or removed from the section key box.

Reference materials (i.e., drug standards) are kept in a locked filing cabinet. Keys for the filing cabinet are assigned to Chief Forensic Chemist and Chief Forensic Toxicologist. The Chief Forensic Chemist may designate other personnel as needed. A logbook of all transactions will be kept and an inventory of all controlled reference materials will be conducted as needed. The return of reference materials to the storage location shall be witnessed by another individual and documented on *DRG-FORM-42 Chronological Log Sheet for Drug Reference Material*.

LOWELL REGIONAL LABORATORY

Each Forensic Chemist has lockable areas to store assigned casework evidence. The back doors at each facility are for maintenance and emergency uses only. These doors shall not be used for daily entrance and exit of the building.

Reference materials (i.e. drug standards) are kept in a locked cabinet. The Chief Forensic Chemist designates key assignment as necessary. A logbook of all transactions will be kept and an inventory of all controlled reference materials will be conducted as needed. The return of reference materials to the storage location shall be witnessed by another individual and documented on *DRG-FORM-42 Chronological Log Sheet for Drug Reference Material*.

6.3.4.2 PREVENTION OF ADVERSE INFLUENCES

The Forensic Chemistry section has multiple measures in place to prevent contamination, cross-contamination, and adverse influence on laboratory activities.

Analysts shall make every effort to maintain a clean, contamination free, workspace. Materials and consumables used in the sampling process and/or analysis shall be stored in closed containers, cabinets, or drawers. These materials include but are not limited to:

- weigh papers/vessels
- pipettes
- test tubes
- vials
- beakers
- capillary tubes
- autosampler vials and caps

Floors and work surfaces should be kept as free of clutter as possible. All surfaces and equipment within the laboratory, where processing and/or analysis occur, should be regarded as potentially contaminated and should be cleaned on a daily basis.

Balances will be checked daily, prior to use, for cleanliness. The weigh pan should be removed and the balance cleaned prior to performance verification for the day. Throughout the day, any visible residue must be removed prior to weighing case samples. Exterior surfaces of equipment to include balances, vortex mixers, microscopes, and heat sealers, should be wiped as appropriate.

Analysts shall ensure that the work area is clean prior to opening an evidence item and after processing each evidence item. Items selected for analysis will be sampled one at a time and sampling materials to include scissors, tweezers, spatulas, and box cutters, will be cleaned with an appropriate solvent between each sampling. Items selected for analysis should be sampled and immediately sealed or maintained in the analysts' short term storage lockers under a temporary seal. A temporary seal shall be such that the contents cannot readily escape. If any evidence is spilled or dropped, the analyst should check the evidence to ensure that all material/units are present. The analyst shall record the occurrence in the case file, and clean the area thoroughly to ensure that contamination of other evidence and the work area does not occur.

General clean-up of work areas should be performed at the close of each work day. In addition to policies listed above, the following schedule should be followed:

- Daily cleaning: bench, equipment, utensils
- Bi-weekly cleaning: test tube racks, vial trays
- Monthly cleaning: lab coats, inner surfaces (drawers, cabinets, areas where consumables are stored), outer surfaces (drawers, cabinets, sink, window sills and chairs)…wipe all surfaces, including partitions, windows, doors and handles
- Bi-annual cleaning: inner storage areas (remove the entire contents of cabinets and drawers), fume hoods, common space areas

One-to-One checks of case number and item numbers shall be conducted when:

- Adding a sample to a container
- Removing a sample from a container and placing it in another container
- Transferring extracted samples for analysis (e.g., TLC plate)

6.3.4.3 SEPARATION

See *ASCL-DOC-01 Quality Manual*.

6.3.5 EXTERNAL ACTIVITIES

See *ASCL-DOC-01 Quality Manual*.

6.4 EQUIPMENT

6.4.1 ACCESS

See *ASCL-DOC-01 Quality Manual*.

6.4.2 OUTSIDE EQUIPMENT

See *ASCL-DOC-01 Quality Manual*.

6.4.3 PROPER FUNCTIONING

The equipment used in Forensic Chemistry is as follows: microscopes, GCMS, FTIR, balances, reference standards, reference materials (RM), reagents, glassware, solvents, and disposable weighing vessels. All purchased chemicals, reference materials/standards, and disposable equipment are considered fit for use when received. If the packaging is damaged or partially opened, the fitness for use will be assessed. Laboratory equipment and instrumentation shall be handled and transported responsibly to ensure optimal performance and to avoid contamination and premature wear/damage.

The specific maintenance and required performance verification for equipment/instrumentation, to ensure proper functioning and prevent contamination or deterioration, is listed within the test method.

OUT OF SERVICE

If an instrument or equipment is not working properly, fails its performance verification, or potential problems are observed, the chemist will immediately take the appropriate steps to repair or correct the problem themselves if they are capable. If the chemist lacks the training or experience to diagnose the problem and restore proper functionality to the equipment, they will clearly mark the equipment 'OUT OF SERVICE' in order to prevent inadvertent use before they seek help in resolving the problem. Maintenance performed to correct the problem must be recorded in the instrument's maintenance log. When it has been determined that instrumentation or equipment was not working properly, the appropriate supervisor shall take into consideration the effect the problem may have had on previous tests. Instrumentation or equipment taken out of service will not be used in casework until appropriate calibration or performance verification is performed.

6.4.3.1 REAGENT RECORDS AND LABELLING

GENERAL

- All purchased solvents, chemicals, reagents, reference materials shall be marked when received with the date and initials of the person receiving them. Upon opening, the bottle shall be marked with the date and initials of the individual opening the substance.

- The quality of all chemicals purchased for use in the Forensic Chemistry Section will be adequate for their intended use. Generally this will mean that solvents, acids, bases, organic and inorganic compounds will be of ACS Reagent Grade or better
- Items with a manufacturer-specified expiration date may not be used after that date without documentation to support continued reliability
- For items without a manufacturer-specified expiration date, dates will be based on experience, industry standard, or scientific consensus
- Appropriate logs are maintained for reagents/chemicals and reference materials used
- Each analyst must ensure that the reference materials, controls, reagents, or chemicals used in their analysis are of satisfactory quality
- Reference materials, controls, reagents, or chemicals which are determined not to be reliable must be removed from use immediately
- The reliability testing shall occur before use or, if appropriate, concurrent with the test
- Preparation (and verification, if needed) instructions are found on the preparation sheet stored in Qualtrax. Recipes may be scaled up or down depending on need.
- Non-routine reagents may be prepared, if the need arises. The preparation shall be documented to the same degree as the routine reagents on their preparation forms, including verification. This documentation is normally recorded in the case notes, as non-routine reagents shall be discarded after use.
- Glassware used for preparation shall be appropriate and clean. Aqueous preparations shall use distilled or E-pure water.
- Only one batch of each type of prepared reagent/chemical will be in use at a time. A batch's date of initial use is the day after preparation, if the previous batch is still in use, and the date of preparation if not. A batch's date of final use is the earlier of either the batch's expiration date or the date of preparation of the subsequent batch. Before a new batch is put into use, the preparer will make sure any excess from the prior batch is discarded. Any exceptions to this will be noted in the appropriate Reagent/Chemical Preparation Book.

CHEMICALS

Preparation: Formulations for preparing routinely used chemicals are located in the *Chemical Preparation Logbook*. Simple solvent mixtures (TLC systems) or acid and base stock solutions will be prepared from materials of adequate quality.

Verification: Prepared chemicals (excluding reagents and reference materials) are not normally subject to additional QC measures.

Labeling: Containers of chemicals will be labeled with:

- identity
- preparation date (if applicable)
- expiration date (if applicable)

TLC systems will only be labeled with the chemical's identity.

Secondary containers to which purchased chemicals are transferred shall be marked with the identity of the contents and the lot number.

Documentation: The *Chemical Preparation Log* must include:

- identity
- preparation instructions
- amount made
- preparation date
- expiration date or expiration time frame
- lot numbers of solvents and/or compounds used in preparation
- initials of the preparer

Controlled forms for all routine chemical preparation logs and recipes are located in Qualtrax.

REAGENTS

Preparation: Formulations for preparing routinely used reagents are located in the *Reagent Preparation Logbook.*

Verification: Verification procedures for routinely prepared reagents are located in the *Reagent Preparation Logbook*. Each new batch of reagent that is prepared must be verified prior to use in casework. Verification may be done by the preparer or by another chemist. The verifier will initial the *Reagent Preparation Logbook* for that batch of reagent to certify that the reagent performed as expected.

Re-verification: Some reagents may be re-verified. If re-verification is an option, those instructions are located on the reagent preparation form.

Labeling: Reagent containers must be labeled with:

- identity
- preparation date
- expiration date

Documentation: The *Reagent Preparation Log* must include:

- identity
- preparation instructions
- amount of reagent made
- preparation date
- expiration date or expiration time frame
- lot numbers of solvents and/or compounds used in preparation
- a method to verify the reagent's reliability (if applicable)

- initials of the preparer and verifier of reagent

Controlled forms for all routine reagent preparation logs and recipes are located in Qualtrax.

REFERENCE MATERIALS

Verification: All reference materials (purchased, prepared, secondary, controlled, non-controlled) shall be verified prior to use in casework. The verification can be achieved by a Certificate of Analysis from the vendor or analyzing the sample via FTIR or GC-MS. All verification information shall be saved in the Quality Records folder in Qualtrax. (Quality Records/Forensic Chemistry/Equipment/Reference Materials)

Secondary Reference Materials (only allowed for qualitative testing)

The collection of reference material from casework shall be documented in the case notes and the agency shall be notified. The secondary reference material shall be recorded in the *Secondary Reference Materials Logbook*; the entry shall include the secondary reference material entry number (SRM#), a unique identifier (i.e., the ASCL case number and item number if it was retained from casework), the method of verification, and the verifier's initials. Containers, for secondary reference materials, shall be labeled with the identity, the secondary reference material entry number, and the unique identifier.

Preparation: Prior to preparation, the preparer shall ensure the reference material has proper verification documentation. The preparer shall document the method of preparation on *DRG-FORM-29 Reference Material Preparation.* The procedure description should contain enough detail to ensure reproducibility.

Labeling: All prepared reference materials shall be labeled with the reference material's identity, designation, the date of preparation, and the expiration date. Prepared qualitative reference materials expire one year after their preparation or on the date specified on the manufacturers' documents/bottle.

Reverification: Prepared qualitative reference materials may be re-verified, after their expiration date, to confirm the reliability of the reference material. The procedure is listed below:

1. Run reference material on GC-MS
2. Both the chromatogram and the mass spectral data shall be evaluated for acceptability for continued use (abundance of the chromatographic peak, presence of any extra peaks that cannot be explained, etc.)
3. Document the reverification and date on the original prep sheet. Record the new reference material designation to include the date of reverification and new expiration date. (e.g., METHA190107 is reverified on 01-09-2020 the new designation would be METHA200109 and the expiration would be 1-9-2021)

All reverification information shall be saved in the Quality Records folder in Qualtrax (Quality Records/Forensic Chemistry/Equipment/Reference Materials) using the naming system: New Designation (reverified from Old Designation). The expiration date shall be set for one year from the re-verification date. In addition to the identity and new reference material designation, the label shall contain the original prep date and the new expiration date.

6.4.3.2 REFERENCE COLLECTION RECORDS

Forensic Chemistry uses reference collection libraries for comparison to known reference materials in the GC-MS, FTIR, and Pharmaceutical Identification testing techniques. Each reference collection has entries documented, uniquely identified, and properly protected.

6.4.4 PERFORMANCE VERIFICATION

Performance verifications shall occur prior to instrumentation being put into service. These performance verifications shall be documented and retained.

New Equipment Performance Verification

Performance verification shall be done and recorded prior to putting instruments/equipment into use.

Microscopes

A plant material sample known to contain cystolithic hairs shall be observed using the new microscope.

GC-MS

After the instrument manufacturer's installation is complete, the following verification shall be performed prior to the instrument being placed into service.

- Add routine methods from instrument with compatible operating software
- Run each routine method with either a test mix of drug reference materials that are commonly identified using that method, or a single drug that is commonly identified with that method
- Evaluate the chromatography, fragmentation patterns, and library matching capabilities to ensure quality performance of the instrument and software
- Retain this data and information in the appropriate location

FTIR

After the instrument manufacturer's installation is complete the monthly performance check for the FTIR shall be performed and saved into the appropriate location. If the performance check passes, the FTIR is ready to be put into service.

A performance verification shall be performed on instrumentation and equipment that has gone outside of the direct control of the laboratory (e.g., for repair or preventive maintenance) to ensure that its calibration status is satisfactory before being returned to service. Instrument logs will reflect that the equipment was functioning properly prior to being returned to service.

6.4.5 FITNESS FOR SERVICE

See *ASCL-DOC-01 Quality Manual*. Also see BALANCES section 9.2 of this document.

6.4.6 CALIBRATION REQUIREMENT

See *ASCL-DOC-01 Quality Manual*.

6.4.7 CALIBRATION PROGRAM

6.4.7.1 COMPONENTS

Listed below is the equipment with its calibration interval. The equipment will be calibrated by a service provider accredited to ISO/IEC 17025 accredited calibration laboratory or replaced after the calibration interval has passed.

Calibration certificates shall contain the measurement results, including the measurement uncertainty or a statement of compliance with an identified metrological specification. These certificates will be located in Qualtrax.

Equipment	**Calibration Interval**	**Tolerance 'as found'**
Balances, Analytical	5 years	1%, safety factor: 2
Balances, Toploading	5 years	5%, safety factor: 2
Balances, Bulk	5 years	5%, safety factor: 2
Traceable weights used for performance checks (multiple masses)	10 years	See table below
Electronic Pipettes (300uL, 1000uL)	Yearly	See table below

Nominal Value - Weight	**Tolerance 'as found'**
10kg	2.0000g
2000g	20.0000mg
100g	0.5000mg
5g	0.0680mg

Nominal volume µL – pipette volume	Maximum permissible error
30 (20-300 µL pipette)	4.0 % (1.2 µL)
150 (20-300 µL pipette)	1.6 % (2.4 µL)
300 (20-300 µL pipette)	0.8 % (2.4 µL)
100 (100-1000µL pipette)	4.0 % (4.0 µL)
500 (100-1000µL pipette)	1.6 % (8.0 µL)
1000 (100-1000µL pipette)	0.8 % (8.0 µL)

Quarterly pipette checks shall be performed and documented. The above criteria shall be used to evaluate the pipette's acceptability. If the pipette falls outside of the acceptable range, potential causes shall be evaluated. The pipette shall not be returned to service until it can meet the above criteria.

6.4.8 LABELLING

See *ASCL-DOC-01 Quality Manual*.

6.4.9 OUT OF SERVICE

See *ASCL-DOC-01 Quality Manual*.

6.4.10 INTERMEDIATE CHECKS

Balances and pipettes are subjected to intermediate checks. These procedures are listed in sections 9.2.4 and 6.4.7 of this manual.

6.4.11 CORRECTION FACTORS

See *ASCL-DOC-01 Quality Manual*.

6.4.12 EQUIPMENT ADJUSTMENT

See *ASCL-DOC-01 Quality Manual*.

6.4.13 EQUIPMENT RECORDS

Records are retained for equipment that influences laboratory activities. These records include the Forensic Chemistry Equipment Log, Calibration Certificates, Reagent Logbooks, microscope cleaning/service records, and instrument and balance logs.

6.5 METROLOGICAL TRACEABILITY

See *ASCL-DOC-01 Quality Manual*.

6.6 EXTERNALLY-PROVIDED PRODUCTS AND SERVICES

If a material or service must meet certain specifications in order to properly function in testing, these items and the required specifications (e.g., manufacturer, type, grade or other technical data relevant to the supply or service) will be communicated to the Procurement Section through a Procurement Request workflow in Qualtrax.

Supplies, reagents, and consumable materials that affect the quality of tests are not used until they have been visually verified to meet the previously-defined specifications. Inconsistencies will be reconciled before materials are utilized in casework.

As chemicals are first opened in the section, the opener is responsible for initialing and dating the container. Supplies, reagents, and consumable materials shall be stored in accordance with the manufacturer's recommendations.

Critical consumables, supplies, and services which affect the quality of testing will be obtained from reliable suppliers.

In the Forensic Chemistry Section, the critical consumables are:

- Certified standards/reference materials
- PFTBA (perfluorotributylamine) GC-MS tuning compound

In the Forensic Chemistry Section, the critical supplies are:

- Certified reference weight for balance adjustment
- Polystyrene reference materials (various forms) for FTIR performance verifications

7 PROCESS REQUIREMENTS

7.1 REVIEW OF REQUESTS, TENDERS, AND CONTRACTS

7.1.1 GENERAL

The Forensic Chemistry Section processes evidence submitted by external law enforcement agencies and the Medical Examiner's Office of the Arkansas State Crime Laboratory. Contracts (submission sheets) are reviewed by Forensic Chemistry personnel to assess the requests made by the customer; if any changes or amendments are necessary all affected personnel shall be notified.

Evidence submitted in death investigation cases is assessed to determine whether the evidence needs processing to aid in the death investigation. The appropriate supervisor may contact the agency or the medical examiner of record for more information, should it be needed.

7.1.2 INAPPROPRIATE REQUESTS

The Forensic Chemistry Section will not routinely process found property. If a case is identified during submission sheet review to fit this description, personnel should turn the submission form over to the appropriate supervisor who may contact the submitting agency to verify that no suspect exists. The supervisor, or designee, may then cancel the request for analysis.

7.2 SELECTION, VERIFICATION, AND VALIDATION OF METHODS

7.2.1 SELECTION AND VERIFICATION OF METHODS

See *ASCL-DOC-01 Quality Manual*. Forensic Chemistry's test methods are listed in Section 9 of this manual.

7.2.2 VALIDATION OF METHODS

See *ASCL-DOC-01 Quality Manual*.

7.3 SAMPLING

All evidence sample containers (i.e, test tubes, beakers, auto sampler vials, etc.) shall be labeled with at least the significant digits of the ASCL case number and the exhibit (item) number.

When practicable, two separate aliquots from an evidence item shall be collected for analysis using separate analytical testing schemes.

7.3.1 GENERAL

For cases containing multiple exhibits, the chemist will select exhibits to test, based on their training and experience, which will substantiate the highest charge the case will support. Items listed as probable cause for a search shall be selected for analysis. Cross contamination of items may preclude the examination of the contaminated items. When practicable, evidence should be left for additional testing, should that be necessary. If the item was a residue or a residual amount is left after obtaining a sample, it is recommended that the GCMS vial be repackaged into the evidence for potential future reanalysis. The case notes shall reflect any inclusion of GCMS vials in repackaging of the evidence.

DEATH INVESTIGATION–OVERDOSE CASES

These cases are generally prioritized and processed as received. Testing on these cases does not always follow typical item selection for analysis and may require additional items be tested. Typically all items with different appearances will be tested. Analysts shall consult a supervisor or tech lead if guidance is necessary.

PURPOSE TO DELIVER FACTORS

There are factors that indicate purpose to deliver that shall be considered when processing evidence. The analyst shall consider which items best support the purpose to deliver charge.

Factors that may indicate purpose to deliver as listed in the Arkansas code:

- The person possesses the means to weigh, separate, or package controlled substances
- The person possesses a record indicating a drug-related transaction
- The controlled substance is separated and packaged in a manner to facilitate delivery
- The person possesses a firearm that is in the immediate physical control of the person at the time of possession of the controlled substance
    - If the AOC CourtConnect record indicates an individual is being charged with a Class Y Felony of simultaneous possession of a firearm and felony drugs – the felony controlled substance may be tested to show presence but not up to a weight threshold. The AOC CourtConnect record shall be indexed into the case record.
- The person possesses at least two (2) other controlled substances in any amount

Guidance on Purpose to Deliver

Weigh, Separate, Package – Scales

In deciding what may be necessary to test, scales with plant material residue or no visible residue shall be considered as a D felony, scales with other residue shall be considered as a B felony.
Use the “purpose to deliver” column on the Drug Matrix Chart

- Scale and weighable drugs (same charge) – test weighable drugs
- Scale and weighable drugs (lower felony) – test both
- Scale and weighable drugs (misdemeanor) – test scale
- Scale and weighable drugs (higher charge) – test weighable drugs

Separated and Packaged
If there are multiple separated and packaged items (more than one) with the same appearance, this may indicate purpose to deliver. If these separated and packaged items are the basis to show purpose to deliver and substantiate the highest charge, one of these items shall be tested, at minimum.

Firearms
- Can assume possession of a firearm by it being a listed item on the drug submission sheet or a separate submission sheet for firearms
- Summary of crime mentioning a firearm – look it up in AOC CourtConnect to confirm this information and include the AOC CourtConnect information

When considering meeting the three separate controlled substances to show intent to deliver, the drug whose schedule and amount achieves the highest charge will be tested to the maximum threshold. Two of the remaining types of scheduled drugs will be tested minimally to show presence only.
Exclusions to the three drug rule are as follows:
- Paraphernalia is not considered
- Illicit tablets that are not pharmaceutical mimics will be considered together
- Partial suspected pharmaceutical tablets may be excluded
- This is not applicable to federal cases
- A Y Felony has been met

No controlled substances or inconclusive results for tests run on selected items will necessitate the testing of additional items until the highest charge possible is substantiated or the supply of evidence items has been exhausted.

7.3.2 SAMPLE SELECTION/COLLECTION

7.3.2.1 SOLIDS (PLANT MATERIAL, POWDER/CRYSTALLINE SUBSTANCE)

Obtain a sufficient portion of the substance for analytical testing. If the substance is in multiple bags, test a portion from enough bags to substantiate the highest charge.

Plants (suspected to be marihuana) are defined as vegetation containing roots, stalks, stems, and leaves. They shall be sampled in the following manner:

1. Note how many plants are present
2. Remove roots

3. Remove small stems and leaves from larger stems and stalk
4. Weigh small stems and leaves
5. Retain a portion for analysis

7.3.2.2 MULTI-UNIT POPULATIONS (PHARMACEUTICAL TABLETS, CAPSULES, OR PARTIAL TABLETS)

The chemist will inspect all the tablets or capsules in an exhibit to ensure consistency and record a count or count by weight, if appropriate, in the case notes. If the item's appearance and imprint code do not indicate the presence of a controlled substance, no further testing is required. Partial tablets can be excluded from testing if they are not suspected to substantiate the highest charge based off of chemist experience.

For items suspected to contain a controlled substance or suspected to be pharmaceutical mimics, a single unit of the population will be tested. The weight of the total population and the weight of the tablet/capsule/partial tablet tested shall be recorded in the case notes and reported.

7.3.2.3 MULTI-UNIT ILLICIT TABLETS

The following scheme shall be followed:

1. Measure weight and assess what may need to be tested
2. Separate, count, and describe groups that need to be tested by shape, color, and imprint
3. Test one tablet, separately, from enough sub items to reach a statutory weight limit based off of the total weight of each sub item OR test one tablet, separately, from a minimum of five sub items.
    a. If the five tested tablets each contain a controlled substance – the analyst may stop and report their findings after appropriate testing is conducted. The report must contain a note, since testing was truncated. (See reporting section for wording)
    b. If the five tested tablets result in a mix of some with controlled substances and some with only non-controlled substances, or if they are all non-controlled/negative – the analyst shall have the data reviewed and contact the prosecutor about how to proceed.
4. All not tested tablets shall be weighed. Depending on the number of remaining tablet types, the analyst may describe as tablets of various shapes, colors, and imprints or separate by shape, color, and imprint if desired.

7.3.2.4 MULTI-UNIT SOLID DOSAGE FORMS (E.G., GUMMY CANDIES, SWEET TARTS, SUGAR CUBES)

A single unit of the population will be tested. The weight of the total population and the weight of the unit tested shall be recorded in the case notes and reported.

7.3.2.5 SOLID DOSAGE FORMS – MULTI– UNIT OR SINGLE UNIT (E.G., BLOTTER PAPER SQUARES, STRIPS, PAPERS)

The weight of total population and the weight of the unit/portion taken for analysis shall be recorded in the notes and reported. If multiple portions are taken for analysis, each portion shall be weighed and analyzed separately. The notes shall reflect whether the portion sampled was returned to the case or destroyed/retained/consumed.

7.3.2.6 PARAPHERNALIA

Paraphernalia is not required to be tested unless:

- It is the only evidence in a case (if multiple items of paraphernalia are present the item that substantiates the highest charged, based on chemist experience, shall be tested)
- It is probable cause
- It can substantiate the highest charge

The notes must indicate whether there is a residue present or no visible residue. A sample from paraphernalia may be obtained by rinsing the item with a suitable solvent.

7.3.2.7 SEEDS

Seeds are not required to be tested unless:

- They are the only evidence in the case
- They are probable cause
- They can substantiate the highest charge

A sample may be obtained by rinsing the seeds with a suitable solvent, or by crushing the seeds and extracting with a suitable solvent.

7.3.2.8 LIQUIDS

Single layer liquids have a reasonable assumption of homogeneity. If appropriate, agitate the liquid well and transfer a portion directly into a screw top vial or covered test tube in order to avoid evaporation of the sample.

7.3.2.9 COMPRESSED ITEMS (PLANT MATERIAL OR POWDERS)

Core sample must be taken if item cannot be broken apart. Notes must reflect how sample is taken (e.g., cored or broken apart).

7.3.2.10 STATISTICAL SAMPLING PLAN FOR MULTI–UNIT POPULATIONS

If statistical sampling is desired to result in testing significantly fewer items, the appropriate supervisor or designee shall be consulted to evaluate the population for homogeneity.

Homogeneity is assessed by comparing all sub-items to each other for visual consistency. If there is a reasonable assumption of homogeneity, a sampling plan may be used. If a sampling plan is employed, the chemist will contact the prosecutor. The chemist will communicate clearly what will and will not be tested, the inference(s) that may reasonably be drawn from the results, and the manner in which the results will be reported.

The Forensic Chemistry section employs the Hypergeometric Distribution sampling plan with a confidence level of 95% and a population interval of 90% (k = 0.9). Determine the number of sub-items (population) that comprises the exhibit. Consult the Hypergeometric Distribution Table below to determine the number of sub-items that must be independently tested.

Sampling Procedure:
Randomly select from the population the number of sub-items determined by the sampling plan. For each sub-item, obtain a portion for analysis and perform necessary tests independently on each sub-item. Clearly label the items that were selected for sampling.

If the tested sub-items return non-homogenous results, all remaining items shall be tested independently until a charge is met, or the prosecuting attorney may be contacted to determine what course of action is required for further analysis.

Hypergeometric Distribution			
Population Size N	**Sample Size***	**Population Size N**	**Sample Size***
5	4	30	17
6	5	40	18
7-8	6	50	19
9	7	60	20
10-11	8	70	21
12-13	9	80	22
14-15	10	90-100	23
16-17	11	101-200	26
18-20	12	300-400	27
21-23	13	500-4999	28
24-27	14	5000-10000	29
28-29	15	>10000	See S:\!FC Controlled Information\Hypergeometric Calculator.xls
* Required sample size to guarantee with 95% confidence that the seizure contains at least a proportion of drugs when k=0.9, if expected that all sampled units contain drugs. If a population size falls in between population sizes listed in the chart, sample for the higher population size.			

7.4 HANDLING OF TEST ITEMS

7.4.1 EVIDENCE STORAGE

7.4.1.1 HANDLING PROCEDURES

See *ASCL-DOC-01 Quality Manual*.

7.4.1.1.1 STORAGE

Forensic Chemistry has a Secure Storage evidence room within the section in the Little Rock facility. This storage area serves as a temporary location for evidence waiting on processing. Personnel within the Little Rock location have fob access to this storage location.

In the Lowell location, Forensic Chemists have access to the location's Secure Storage due to the size of the laboratory. The Forensic Chemists have access by fob during working hours (8-4:30 weekdays), but need a key for entry outside of those hours.

In Little Rock and Lowell, each analyst has their own personal storage area to secure evidence in their absence. The Little Rock location has a storage location to which a key is assigned out to only one chemist at a time when necessary to store bulky items.

If it is necessary to reassign evidence already in a chemist's possession, the supervisor may retrieve the evidence from the analyst's storage area(s). The supervisor will then reassign and transfer the evidence to another analyst. The chain of custody will reflect all transactions.

The supervisor or designee may allow access to an absent analyst's storage area(s) for inventory purposes. The storage area(s) will be locked immediately upon completion of the inventory.

7.4.1.1.2 PACKAGING AND SEALING

If a packaging deficiency is not apparent until the case is checked out by an analyst, the analyst may correct the deficiency. If there is any concern that the packaging deficiency has affected the integrity or identity of the test item, the analyst's supervisor and the customer agency shall be advised and consulted for further instructions. All remedial actions taken to correct packaging or evidence deficiencies shall be noted in the case record (e.g., submission form or analyst's notes).

It is the responsibility of the analyst to maintain proper control of all evidence in their possession. Evidence may be stored for a short or long period of time in the hood when drying is necessary or when there are safety concerns.

7.4.1.2 CRIME SCENE EVIDENCE (ILLICIT LABS)

The following policies and procedures apply exclusively to the Little Rock and Lowell laboratories. The Hope laboratory does not accept evidence associated with controlled substance manufacturing cases (with the exception of marihuana grows).

Illicit Laboratory chemists do not collect evidence from crime scenes for submission to the laboratory. If the ASCL is called to the scene of a clandestine laboratory, our role is to help the on-scene officers know what evidence is appropriate to collect to support manufacturing.

7.4.1.3 RENDERING HAZARDOUS MATERIALS SAFE

Evidence Receiving Technicians may call Forensic Chemistry to render materials safe if they are unsure if the packaging is adequate. The Forensic Chemist shall inspect the packaging and repackage any items that need to be rendered safe. This inspection/repackaging shall be documented on *Illicit Laboratory Evidence Safety Form ER-FORM-01*.

Hazardous materials that could be submitted include: organic powders, inorganic powders, organic solvents, strong aqueous bases, strong aqueous acids, pyrophoric[1] metals, noxious gases, flammable vapors, potent physical drugs (e.g., fentanyl and analogs).

7.4.1.3.1 PACKAGING OF VOLATILE CHEMICALS

If the chemical evidence consists of liquids, these liquids should be packed in a glass vial with a Teflon seal, and the glass vial should be placed in a high density non-reactive plastic bottle. Any evidence that emits acidic, basic, organic, or otherwise dangerous fumes that cannot be trapped in the containers specified above shall not be accepted into the Arkansas State Crime Laboratory evidence receiving section.

7.4.1.3.2 PACKAGING OF HAZARDOUS SOLIDS

Any solid sample that the chemist determines to have hazardous properties should be placed in a glass vial with a Teflon seal and sealed in a high density non-reactive plastic bottle.

7.4.1.3.3 PACKAGING OF IODINE

Iodine shall be packaged with great care to prevent cross contamination. Because of the sublimation properties of iodine, only a small amount (e.g., 1-2 grams) of sample is necessary. It should be packaged in a glass vial with a Teflon seal. The glass vial should then be packaged in a high density non-reactive plastic bottle. Samples of suspected iodine will permeate through most plastic bags and all textile based packaging.

[1] Liable to ignite spontaneously on exposure to air

7.4.1.3.4 PACKAGING OF LITHIUM OR SODIUM METAL

Lithium and sodium metal are pyrophoric upon contact with water and should be handled with extreme caution. Lithium or sodium samples should be stored in a heavy organic solvent or petroleum distillate. No alcohol, ether, acetone, or ketone of any kind should be used to store lithium or sodium. A small amount of lithium or sodium (e.g., one inch square) should be placed in a glass vial with a Teflon seal. An organic solvent (e.g., hexanes, petroleum ether, Coleman fuel, camp fuel) should be poured into the glass vial. It is important to keep the surface of the solvent well above the lithium or sodium metal.

7.4.1.3.5 PACKAGING OF ANHYDROUS AMMONIA

Anhydrous ammonia is a very dangerous basic gas. No anhydrous ammonia containers shall be submitted to the Arkansas State Crime Laboratory. If analysis of ammonia is requested, a small amount of ammonia should be bubbled through deionized water. All handling of anhydrous ammonia containers should be done observing safety standards approved by OSHA and the EPA.

7.4.1.3.6 PACKAGING OF SHARPS

Any evidence that is sharp enough to puncture the skin shall be stored in a puncture proof container.

7.4.1.3.7 PACKAGING OF POTENT PHYSICAL DRUGS

Suspected potent drugs shall be packaged in a manner that the drug cannot escape from the packaging.

7.4.2 ITEM IDENTIFICATION

See *ASCL-DOC-01 Quality Manual*.

7.4.3 DEVIATIONS

If the analyst discovers an inconsistency between the submitted evidence and the submission sheet, or if there is doubt about the suitability of an evidence item for testing, then the analyst shall attempt to contact the customer before proceeding. All contacts will be documented in the case record (e.g., using an *Agency Contact Form* (ASCL-FORM-06), by email). For minor inconsistencies, the analyst shall use their judgment on whether to contact the customer. Plainly visible not listed items do not require agency contact and will not be tested.

Examples that require contact with the agency:

- Missing evidence
- Item description or agency identifier does not conform to the information provided
    - Green plant material is listed, but crystalline substance was received
    - Extra suspect listed on the packaging
    - Suspect name is substantially different between submission sheet and packaging

- Unexplainable weight discrepancy between the submission sheet and the weight obtained
- Substantial difference in agency case number between submission sheet and packaging
- Hidden evidence not listed on the submission sheet (whether being tested or not)

Should contact not be made within five business days, the evidence may be returned to the investigating agency to correct. Information regarding the reason for return shall be attached to the evidence.

7.5 TECHNICAL RECORDS

7.5.1 CASE NOTES

The analyst shall create a set of case notes for each case they analyze. The case notes may be handwritten, typed, or a combination of the two. This section outlines what must be included in the case notes. Any deviation from these guidelines must have approval of the supervisor or designee.

REQUIREMENTS FOR NOTES AND OBSERVATIONS

- Handwritten notes and observations must be in ink. However, pencil may be appropriate for diagrams or making tracings.

- Nothing in the handwritten notes will be obliterated, erased, or deleted. Any corrections made to handwritten notes will be made by an initialed, single strikethrough (so that what is stricken can still be read).

- Both the chemist's and trainee's handwritten initials must be present on each page of the case notes in cases in which a trainee assisted the chemist. Electronic equivalent can be substituted for chemist's handwritten initials. If all of the analysis was not performed by the trainee, it must be clear who was responsible for each activity.

- Each page of the case notes must include:
  - The unique ASCL case number (YYYY-000000)
  - The date(s) the notes were taken on (Should the sampling of a case take longer than one day, it shall be properly noted which day the sampling was resumed.)

- Every evidence item shall have an adequate description explaining the appearance of the item and its packaging. The description shall be detailed enough so that the chemist could identify the evidence based only on their notes. If evidence is not tested, it must be clearly documented.

If there are multiple suspects listed on the submission sheet, any names on inner packaging should be recorded in notes.

- Measurements taken must be documented in the case notes and clearly identifiable to the item to which it corresponds. Brackets shall be placed around the portion of the item and packaging that is included in any gross weights.

- If statistical sampling is employed, it must be clearly documented in the case notes and follow the statistical sampling plan within this manual.

- During testing, the chemist must document in their case record:
  - the tests performed (and by whom, if applicable)
  - the date on which the tests were performed unless supporting examination documentation is marked with the testing date
  - a description and date of any solvent extraction procedure (This excludes tests only for informational purposes like Marquis or Cobalt Thiocyanate to determine possible concentration)
  - the results of the tests
  - non-routine items:
    - re-running a test: how the sample was treated differently, if applicable (e.g., concentrated sample down, re-extracted to prepare more concentrated sample, spotted more heavily), or why the sample was re-run (better chromatography)
    - non-conforming results – state why the results are non-conforming (e.g., no test mix run, failed autotune, bad blank, wrong solvent vial run, etc.)
    - no controlled substance results – shall provide enough information on extraction method/concentration to ensure enough testing has been conducted to support conclusion

- Any other notations required for an individual testing technique are described within this manual with the test method.

7.5.1.1 TECHNICAL RECORD RETENTION

See *ASCL-DOC-01 Quality Manual*.

7.5.1.2 ABBREVIATIONS

Each chemist shall use the secure **Forensic Chemistry Abbreviation Definition List** for any abbreviations used in note taking that are specific to the laboratory. This list shall be available to all forensic chemists and can be found on S:\!FC Controlled Information\forensic chemistry abbreviation definition list. A forensic chemist can add to this list at any time by submitting a new abbreviation to their supervisor or designee. A Forensic Chemistry supervisor or designee shall

review the abbreviation suggestion for overlap and need before adding it to the secure Forensic Chemistry Abbreviation Definition List. Once the new abbreviation definition is added to the secure Forensic Chemistry Abbreviation Definition List, it may be used by any forensic chemist in their note taking process.

7.5.1.3 TECHNICAL RECORD SUFFICIENCY

See *ASCL-DOC-01 Quality Manual*.

7.5.1.4 TECHNICAL RECORD PERMANENCY

See *ASCL-DOC-01 Quality Manual*.

7.5.1.5 REJECTION

If data, an observation, or a calculation is rejected, the following information will be recorded in the technical record:

- The reason for the rejection
- The identity of the person rejecting
- The date of the rejection

This applies to pre-draft complete items as well as rejections in case review.

Examples of when to reject data include:

- Non-conforming data
  - Bad QA/QC, bad blank
  - DP failed
  - Wrong solvent vial run
  - No RM spotted on TLC plate
  - Samples containing compounds with same Rf factor on TLC (e.g., meth/MDMA, Δ8-THC/Δ9-THC)
- Conflicting/inconsistent results
  - Run 1—GCMS: indicative meth, positive THC; Run 2 (concentrated sample)—GCMS: positive meth, THC not detected. The result of THC should be questioned and rejected based off the presence of THC not being confirmed with a more concentrated sample preparation. If the presence of THC cannot be explained – consult supervisor/tech lead as an additional sample may need to be taken to investigate.
- Incorrect results
  - Ran incorrect vial
  - Δ8-THC in 1st run that does not persist after liner change (reject first run)
  - Syringe was found to be clogged and you had sample runs that were blank
  - Psilocin that does not persist with an acetoniltrile extraction (reject first run)

7.5.2 AMENDMENTS TO TECHNICAL RECORDS

Amendments[2] to technical records must be trackable to previous versions or to original observations. Both the original and amended data/files will be retained, including:

- The date of alteration
- An indication of the altered aspect(s)
- The personnel who made the alteration(s)

Any corrections made to existing hardcopy technical records will be made by an initialed and dated single strikeout (so that what is stricken can still be read) by the person making the change. All additions will be initialed and dated. Correction fluid or correction tape may not be used.

Contemporaneous[3] revisions to technical records are not considered to be amendments.

7.6 EVALUATION OF MEASUREMENT UNCERTAINTY

The Forensic Chemistry Section has measurement of uncertainty estimates for the following activities:

- weight determination on the analytical, toploading, and bulk balances (Little Rock and Lowell)
- quantitative determination of THC

The budgets are reviewed/recalculated on a yearly basis, at minimum, and when there are significant changes to procedures, or equipment has undergone calibration. The budget for weighing devices shall be updated and/or recalculated when new equipment is put into service. The budget for quantitative uncertainty shall be updated and/or recalculated when new equipment or a new analyst is added to the procedure.

The current revision of MU budgets is retained in Qualtrax and shall be used. The requirements for measurement uncertainty reporting are addressed separately within this manual.

7.7 ENSURING THE VALIDITY OF RESULTS

7.7.1.1 VERIFICATION

The Forensic Chemistry Section does not perform verification of independent examinations.

[2] Including additions, deletions, changes, interlineations, or any other modification to the original information
[3] Contemporaneous means at the same period of time. Amendments made after moving on to the next case are not considered to be contemporaneous. Amendments made before moving on to the next case, while the matter is still fresh in memory, may be considered contemporaneous.

7.7.1.2 CASE REVIEW

All cases will be technically and administratively reviewed prior to the release of the report. The review process must confirm that electronic versions of all necessary documentation are in the imaging module of the JusticeTrax LIMS-plus program. Each technical and administrative review will cover, at minimum, the items listed on *ASCL-FORM-05* and ensure the evidence has been returned to a secure storage location.

If a reviewer discovers an error in the case record, the reviewer must document the error, their initials, and the date in the *Reviewer Notes* field, in the related request in JusticeTrax, and inform the analyst. If the analyst and reviewer cannot reach a consensus, then both the analyst and reviewer must meet with a supervisor or the technical lead for resolution.

If the error requires the analyst to correct administrative and/or examination records, the original record will remain in the electronic case file and the corrected record stored with a different name (e.g., corrected notes, corrected data, etc.). If there is a change to the report, the original report should be marked (e.g., "Draft Report", "Original Report) and scanned into the JusticeTrax case file.

Sometimes bigger cases will require two reviewers. Once each reviewer has completed the review, they will meet and record the errors, their initials, and the date in the *Reviewer Notes* field in JusticeTrax and inform the analyst. The completion of the review should be recorded by one reviewer rolling the tech review milestone and the other reviewer rolling the administrative review milestone.

7.7.1.2.1 TECHNICAL REVIEW

See *ASCL-DOC-01 Quality Manual*.

7.7.1.2.2 ADMINISTRATIVE REVIEW

See *ASCL-DOC-01 Quality Manual*.

7.7.1.2.3 TESTIMONY REVIEW

The Forensic Chemistry Section ensures that analysts are accompanied and reviewed by an experienced analyst on their first testimony. Should improvements be needed, they may be re-reviewed on future testimonies to ensure they are effective. In addition, each analyst shall be reviewed once per accreditation cycle, when practicable.

7.7.2 INTERLABORATORY COMPARISONS

See *ASCL-DOC-01 Quality Manual*.

7.7.3 MONITORING ACTIVITY ANALYSIS

See *ASCL-DOC-01 Quality Manual*.

7.7.4 INDIVIDUAL PERFORMANCE MONITORING

See *ASCL-DOC-01 Quality Manual*.

7.7.5 PERFORMANCE MONITORING REQUIREMENTS

The Forensic Chemistry Section follows the policy in *ASCL-DOC-01 Quality Manual* proficiency testing/intralaboratory comparisons. Test specimens may be obtained from external providers or prepared internally. Intralaboratory comparisons (Internal proficiency tests) may include previous external proficiency samples, samples retained from casework (secondary proficiency reference materials), samples prepared from primary reference materials, re-examination techniques, and blind techniques.

The Forensic Chemistry Section uses the following criteria to evaluate the results of proficiency tests/intralaboratory comparisons.

MASS DETERMINATION EVALUATION

A mass determination component may be included as a part of a proficiency test. Tests that include this component are determined prior to assignment.

SATISFACTORY: Mass range is ± 5mg from preparation measurement. Any sample loss (e.g. static, transfer loss) may cause variation from this range.

Supervisor evaluation is required if reported measurement is outside of the expected range.

UNSATISFACTORY: Mass range is outside of expected range with no justification.

QUALITATIVE PROFICIENCY TEST EVALUATION

SATISFACTORY: Identification of all expected controlled substances and general chemicals/non-controlled substances

UNSATISFACTORY: Incorrect or incomplete identification of expected controlled substances and general chemicals/non-controlled substances

QUANTITATIVE △9–THC ANALYSIS EVALUATION

SATISFACTORY: The average of the two results is within 20% relative difference from the expected value or within two standard deviations of the consensus mean

UNSATISFACTORY: The average of the two results is not within 20% relative difference from the expected value or not within two standard deviations of the consensus mean

CLANDESTINE LABORATORY ANALYSIS EVALUATION

SATISFACTORY: Correct identification of all controlled substances, non-controlled substances, and/or elements expected

UNSATISFACTORY: Incorrect or incomplete identification of all controlled substances, non-controlled substances, and/or elements expected

7.7.6 PERFORMANCE MONITORING SCHEDULE

The proficiency testing schedule is maintained by the Section Chief and/or the Technical Leader and is available on the shared drug drive.

7.7.7 PROFICIENCY TEST SOURCING

See *ASCL-DOC-01 Quality Manual*.

7.7.8 PERFORMANCE MONITORING RECORDS

See *ASCL-DOC-01 Quality Manual*.

7.7.9 RE-EXAMINATION POLICY

The Forensic Chemistry casework re-examination policy is a quality assurance policy that is intended to evaluate the reliability of our analytical results and the competency of our analysts. This is achieved by internal comparison of results for cases chosen for re-examination.

Re-examination shall be performed by a second competent analyst. Only the items originally analyzed will be re-analyzed. The evidence will be re-examined using appropriate procedures and test methods.

All re-examination data, notes, and other documentation shall be contained within the original case record under the "Re-examination Request." The original analyst's name (Last, First) shall be recorded in the "Requestor" box located on the Request Tab in Justicetrax.

Results of both testing activities shall be compared by the analyst performing the re-analysis. Any discrepancies shall be evaluated. Discrepancies that cannot be explained shall require investigation by the Chief Forensic Chemist and/or Technical Leader.

While the main focus of casework re-examination is the comparison of original analytical result(s) to re-examination results, the re-examination should also include the evaluation of some, or all, of the following:

- that the original descriptions were adequate and complete
- that all seals made by the original analyst are appropriate and properly labeled
- that the item(s) and outer packaging were properly sealed to prevent contamination or deleterious change
- that all counts and weights are consistent with original documentation

- if applicable, that the analyst correctly selected items to test to substantiate the potential highest charge based on the appearance of the items and/or additional information listed on the inner packaging (e.g., evidence listed with a specific suspect's name or date of offense/buy date)
- if applicable, that the notes reflect any repackaging and/or inclusion of GC/MS vials

All conflicting results and/or observations shall be evaluated to determine the level of significance of the discrepancy. If necessary, a Quality Assurance Concern (QAC) shall be initiated.

Any nonconformities, deficiencies, or departures from accepted laboratory or section standards identified during the course of re-examination shall be documented and may require initiation of a QAC. If a QAC isn't required, the documentation will be maintained by the Chief Forensic Chemist and/or Technical Leader. Due to the wide variability of Drug casework and potential occurrences necessitating action, a different degree of response may be required from one instance to another. If retraining or remedial actions are necessary, an action plan will be developed based on the nature of the cause. Actions taken may include, but are not limited to, observation-based casework monitoring, removal of authorities/responsibilities, re-examination of additional casework, and/or other actions as deemed necessary.

Cases undergoing re-examination, as part of the quality assurance program, shall have a re-examination letter issued. The letter shall state "This case was selected for re-examination as part of our quality assurance program. If this re-examination necessitates a change to the original report of analysis, an amended report will be issued."

An amended report is necessary when:

- original reported results are incorrect
- there is an error on the original report that needs correction

7.8 LANGUAGE FOR REPORTS AND TESTIMONY

7.8.1 GENERAL

See *ASCL-DOC-01 Quality Manual*.

7.8.1.1 DOCUMENTATION

See *ASCL-DOC-01 Quality Manual*.

7.8.1.2 REPORTS

An ASCL "Report of Laboratory Analysis" is generated at the conclusion of analytical testing. These reports normally consist of administrative information in a "header" and technical information in

the report body. For each item listed the report body contains three columns of information: “Items”, “Evidence Description” and “Test Results”. When applicable, the body may also include statements about evidence sampling or disclaimers that aid the customer in understanding the report.

If there were multiple submissions, on the same case, from separate investigating officers, a report shall be issued to each officer containing the evidence they submitted for analysis.

The results of testing carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively. This document describes general guidelines intended to cover reporting of the majority of cases analyzed. However, situations may arise which require deviation from these guidelines due to the extreme variability of evidence received. In such a case, the chemist will consult the supervisor to determine an approved method for reporting the information.

Upon completion of the report, the chemist shall review the header information (e.g., investigating officer/agency/address, suspect(s), victim(s), laboratory case number, agency case number, etc.) and the analytical results (item numbers, item descriptions, test results, weights, etc.) reported to ensure they are correct. Once proofing is complete, the analyst will sign the report by marking the request ‘Draft Complete’ in JusticeTrax.

7.8.2 COMMON REQUIREMENTS FOR REPORTS

7.8.2.1 REPORT ELEMENTS

For a broad list of elements included and not included in the report see *ASCL-DOC-01 Quality Manual*.

7.8.2.1.1.1 GUIDELINES FOR ALL ITEMS RECEIVED

All items (exhibits) submitted, shall be included on the report.

ITEMS LISTED ON SUBMISSION SHEET

The following shall be present for all reported items:

- The item (exhibit) number
- A description of the item including, where appropriate, count/count by weight
    - Containers are not required in report descriptions with the exceptions of:
        - Items where reported gross weight includes any packaging
        - Paraphernalia (smoking device, grinder, syringe, vape pen/cartridge, etc.) containing weighable substances
- The exhibit’s net or gross weight (in grams or kilograms) with units and measurement uncertainty with exceptions of:

    - Items for which a weight is not required to be taken by this manual
    - Items that contained a residue or no visible residue: report residue, where appropriate
    - Items that weigh less than 50mg: report as "less than 50mg" with no measurement uncertainty
    - Non-controlled pharmaceutically identified tablets
- The result(s) of analytical testing, if it was conducted (see guidelines for reporting items that were tested)
- The result of "not tested" for items not undergoing testing
- A disclaimer about items undergoing partial testing, chemist assignment of item numbers with reason, or relatable discrepancy in item number(s)

ITEMS (OF EVIDENTIARY VALUE) NOT LISTED ON THE SUBMISSION SHEET

Not listed items shall be listed at the bottom of the report. No weight is required to be reported on these items (but it must be recorded in the notes).

> Example: NOTE: "The following items were not listed on the submission form and received no analysis: one straw, one plastic bag, and one plastic bag containing green vegetable material."

7.8.2.1.1.2 GUIDELINES FOR ITEMS THAT WERE TESTED

GENERAL

- Salt/base form or diastereomer may be reported based on a positive FTIR test
- If testing positively identifies a set of stereoisomers in an item but does not positively identify the specific stereoisomer present, the results may be reported as *stereoisomer a/ stereoisomer b* (e.g., pseudoephedrine/ephedrine, zopiclone/eszopiclone, citalopram/escitalopram)
- Items that break down during testing to another substance that is the same schedule/charge or items that cannot be distinguished between with our testing shall be reported as follows:
    - psilocyn/psilocybin
    - gamma-butyrolactone (GBL)/gamma-hydroxybutyric acid (GHB)
- If no element, compound, or substance was positively identified, the results may be reported as "no controlled substances detected"
- For items that were ***color tested only*** the following disclaimer (or a rewording with similar language to include multiple items/tablets/capsules) shall be on the report, "A color test was performed to screen for the presence of various drug classes and/or organic functional groups"

TABLETS/CAPSULES and SEALED PHARMACEUTICAL PRODUCTS

- Both count and weight shall be reported for tablets or capsules tested or identified to contain a controlled substance

- For items pharmaceutically identified to contain non-controlled substances that receive no further analysis, the active ingredients, and if desired the dosage, may be reported.
  If only non-controlled substances are present in the case, the pharmaceutical identification shall be reported. The result shall be preceded by the phrase “identified as*” and the report shall contain the following disclaimer “*The identification results were obtained by comparing the item to reference sources and not by analytical testing. Any results confirmed by analytical testing are listed separately.”
- For items pharmaceutically identified to contain a controlled substance, that breaks down on the GC-MS to another drug or does not chromatograph well, the active ingredients, and if desired the dosage, shall be reported if the substance substantiates the highest charge or is the third drug to show purpose to deliver. The result shall be preceded by the phrase “identified as*” and the report shall contain the following disclaimer “*The identification results were obtained by comparing the item to reference sources and not by analytical testing. Any results confirmed by analytical testing are listed separately.”
- For items pharmaceutically identified to contain a controlled substance, items that were indicative or negative for pharmaceutical identification, or non-controlled items with positive pharmaceutical identification where no “identified as*” result was reported where no other analysis was conducted, the following disclaimer shall be on the bottom of the report. “Tablets were compared to reference sources.”

MULTI-UNIT POPULATIONS

The report must state what was submitted, what was tested, and must be clear that the result/conclusion pertains only to what was tested. Illicit tablet cases in which testing was truncated without contacting the prosecutor shall contain the following note. "If more testing is necessary for this case, please contact a Drug Section supervisor and your request for additional testing will be prioritized."

- For multi-unit populations composed of solid dosage forms (e.g., LSD on blotter paper squares, gummies, tablets, partial tablets, capsules, sublingual films, etc.)
    - A description (which includes the word "submitted") of the entire population including a gross weight/net weight and identity of dosage form (if known)
    - A description (which includes the word "tested") of what was tested, the total net/gross weight of the items tested, and the results

- For all other multi-unit populations, when reported together and only a portion of the population is tested, the report may include in some variation:
    - A description (which includes the word "submitted") of the entire population including a gross weight/net weight
    - A description (which includes the word "tested") of what was tested, the total net weight of the items tested, and the results
    - A description (which includes the words "not tested") of what was not tested, the total gross weight/net weight of the items not tested

SEMI-QUANTITATIVE RESULTS FOR PLANT MATERIAL TESTING

Plant material with response ratio greater than or equal to 1 and criteria for reporting marihuana have been met:

- Report the appropriate result in the Justice Trax module to display the desired result and the following disclaimer. [1] "As determined by comparison with a 1% Δ9-tetrahydrocannabinol standard."

Plant material with response ratio less than 1 and criteria for reporting Δ9-THC have been met:

- Report the appropriate result in the Justice Trax module to display the desired result and the following disclaimer. [2] "The response of this sample was less than that of a 1% Δ9-tetrahydrocannabinol standard. If quantitation is necessary, please contact the laboratory for further analysis."

Plant material with a negative microscopic test with response ratio greater than or equal to 1 and criteria for reporting Δ9-THC have been met:

- Report the appropriate result in the Justice Trax module to display the desired result and the following disclaimer. [3] "The response of this sample was greater than or equal to that of a 1% Δ9-tetrahydrocannabinol standard."

Δ9-THC QUANTITATIVE DETERMINATION

The report shall be clear about what portion of the sample received quantitative determination.
Example (description wording may be changed based off of sample type or analyst preference):

Submitted: green plant material/green vegetable material (report net weight of entire item with appropriate measurement uncertainty)

Tested: portion of green plant material / green vegetable material (report total net weight tested in grams w/ appropriate uncertainty, and the average of two quantitative determinations truncated to two significant figures, Δ9-THC, and appropriate quantitative measurement uncertainty)[4] other compounds identified may also be reported

For samples with averages falling below 0.10%, the results shall be reported as < 0.10% Δ9-THC with no measurement uncertainty. For samples with averages falling above 1.0%, the results shall be reported as > 1.0% Δ9-THC with no measurement uncertainty.

All Δ9-THC quant results shall have the following disclaimer reported:

"[a] The reported quantitative result is the total measured Δ9-tetrahydrocannabinol (Δ9-THC) by percent dry weight. Total Δ9-THC is the sum of Δ9-THC and Δ9-tetrahydrocannabinolic acid (Δ9-THCA)."

MANUFACTURING CASES

- If only elements or non-controlled substances were positively identified in an exhibit, the chemist may report "no controlled substances detected" with or without the positively identified elements or substances
- The following table outlines the proper method to report results which may be significant in the manufacturing process.

[4] For example, 0.51% ±0.11% Δ9-THC

Reporting Results Significant to Manufacturing Cases	
Result Reported	**Necessary Tests**
Phosphorus/Iodine	XRF
Inorganic salts (e.g., Ammonium nitrate, Sodium phosphate etc.)	IR solid
Ammonia[1]	IR vapor, Nessler's
Lithium[2]	IR solid, Flame Test
Lithium metal	IR solid, Flame Test, Reactive w/ H_2O
Sodium[3]	XRF, IR solid
Sodium metal	XRF, Reactive with H_2O
Other elements[4]	XRF alone, or IR and Flame Test
Acidic solution/basic solution	pH test
[1] The following disclaimer must be added between the report's last evidence item and the analyst's signature: "The presence of ammonia does not confirm the presence of anhydrous ammonia." [2]The following disclaimer must be added between the report's last evidence item and the analyst's signature: "The presence of lithium does not confirm the presence of elemental lithium." [3] The following disclaimer must be added between the report's last evidence item and the analyst's signature: "The presence of sodium does not confirm the presence of elemental sodium." [4] The following disclaimer must be added between the report's last evidence item and the analyst's signature: "The presence of "*insert element*" does not confirm the presence of elemental "insert element"."	

7.8.2.1.1.3 GUIDELINES FOR ITEMS THAT WERE NOT TESTED

The report shall clearly communicate which items (or portions of items) were not tested.

7.8.3 SPECIFIC REQUIREMENT FOR TEST REPORTS

7.8.3.1 ADDITIONAL STATEMENTS

See *ASCL-DOC-01 Quality Manual*.

7.8.3.2 REPORTING SAMPLING

If the Hypergeometric Distribution sampling plan is used, the report for the item must also include a statement indicating the confidence level and population interval specified by the plan.

7.8.4 SPECIFIC REQUIREMENTS FOR CALIBRATION CERTIFICATES

See *ASCL-DOC-01 Quality Manual*.

7.8.5 REPORTING SAMPLING – SPECIFIC REQUIREMENTS

Please refer to Section 7.8.3.2.

7.8.6 REPORTING STATEMENTS OF CONFORMITY

See *ASCL-DOC-01 Quality Manual*.

7.8.7 REPORTING OPINIONS AND INTREPRETATIONS

See *ASCL-DOC-01 Quality Manual*.

7.8.8 AMENDMENTS TO REPORTS

7.8.8.1 IDENTIFYING THE CHANGE(S)

An amended report is necessary if an error is found on an issued report (including reports uploaded to iResults). An "amended request" will be created in the LIMS and all administrative and examination records for the amended analysis will be added to the electronic case record. Administrative and technical reviews are required before an amended report is issued. The Section Chief or Technical Lead will review all amended requests. If the amended report is necessitated due to an error missed by the original reviewer, they may be included in the amended review process. Should two reviewers be needed, documentation of this review will be incorporated into the original case file, by each reviewer rolling one of the milestones on the Amended Request.

The original report and all original records will be kept in the case record.

When an amended report is issued, any change of information will be clearly identified. Where appropriate, the reason for the change will be included in the report.

7.8.8.2 STYLE OF AMENDMENT

See *ASCL-DOC-01 Quality Manual*.

7.8.8.3 IDENTIFYING THE AMENDED REPORT

The statement "AMENDED REPORT TO ORIGINAL [TYPE] REPORT ON [DATE]" (or equivalent) will appear below the header information and above the listing of the evidence and the results. The date of the original report must be entered in the "additional data" tab of the amended request. The amended report will contain all of the items on the original report and any amendments

7.8.9 SUPPLEMENTAL REPORTS

A supplemental report is necessary when additional evidence is received after the original report has been issued, additional requests for analysis are made, or other additional testing is required in a case (Note: When additional evidence is received on a case that has not been completed, the additional evidence may be analyzed and included in the original report.)

Evidence resubmitted to the laboratory for testing shall be inspected by the analyst to ensure it is in substantially the same condition as when the analyst completed the original analysis. Items not undergoing additional testing may be described only. If the case has left the control of the laboratory, a new weight shall be recorded in the notes, for items undergoing additional testing, regardless of whether it is necessary for reporting. If the analysis associated with a supplemental request is completed by the original chemist, original weights must be used on the supplemental report. Only items undergoing additional testing will be included in the supplemental report.

If the analysis associated with a supplemental request is completed by a chemist other than the one assigned to the original request a supplemental report is necessary if ONLY new items are tested.

If reanalysis of previously tested items occurs (this excludes the quality assurance re-examination program) the request type shall be a new request not a supplemental. The report shall contain a note including date(s) of previous report(s). Example: Reanalysis of items previously reported on Drug Report issued 06/22/2020 and Supplemental Drug Report issued 7/21/2020.

7.8.10 LANGUAGE FOR TESTIMONY

Forensic Chemistry analysts may be called to testify to their conclusions in a court of law. The analyst shall testify without bias and stay within the boundaries of their area of expertise.

An analyst may testify to any of the following:

- Education, training, experience
- Results of analytical testing
- Conclusions from analysis
- Weight of sample and associated measurement of uncertainty
- Analytical testing scheme and theory of how the tests work

7.8.10.1 DEFINITIONS OF CONCLUSIONS

IDENTIFICATION OF SUBSTANCE THROUGH ANALYTICAL TESTING

The substance has been identified through appropriate analytical testing. The analyst may testify to the conclusion of the substance being present in the sample analyzed.

IDENTIFICATION OF SUBSTANCE THROUGH PHARMACEUTICAL IDENTIFICATION

Analyst may testify that physical characteristics and imprint on the submitted evidence were consistent with the pharmaceutical reference source. They may make no assertions as to what is actually in the item unless separate analytical testing was conducted.

NOT IDENTIFIED (NO CONTROLLED SUBSTANCES DETECTED)

Analyst may testify that we were unable to detect or confirm any controlled substances through the analytical testing performed.

SEMI-QUANT RESULTS (This testing is indicated on the report of analysis by superscript 1,2, or 3 in front of the reported results and should contain a disclaimer with the same number)

Marihuana – Analyst may testify that the sample is marihuana if the appropriate testing scheme has been followed. (See 9.1.1.2) The analyst may state whether the response of the sample was greater than or equal to the response of the 1% Δ9-THC standard. The analyst may not make any statement on the potential Δ9-THC concentration in the sample.

Δ9-THC – Analyst may testify to the components detected in the sample if the appropriate testing scheme has been followed. (See 9.1.1.2) The analyst may state whether the response of the sample was greater than, less than, or equal to the response of the 1% Δ9-THC standard. The analyst may not make any statement on the potential Δ9-THC concentration in the sample.

MANUFACTURING

An Illicit Lab chemist can testify to substances that meet the requirements for reporting. They may provide their opinion on the manufacturing method.

Analysts may not testify to manufacturing in plant material cases.

7.8.10.2 LIMITATIONS OF DRUG ANALYST TESTIMONY

- If a substance is identified in casework evidence, the analyst shall make no assumption or suggestion as to the source of the evidence, how that substance was transferred to the evidence, or how long that substance has been present in the evidence.
- When analyzing a portion of a population, an analyst shall not state their conclusion applies to the entire population (or a percentage of the population) unless statistical sampling was employed. When statistical sampling is employed, the analyst shall clearly

explain the conclusion being made, the results of the sampling units tested, and the confidence level.[5]

- An analyst shall not state that drug chemistry examinations are infallible or have a zero error rate.
- An analyst shall not provide a conclusion that includes a statistic or numerical degree of probability except when based on relevant and appropriate data.
- An analyst shall not cite the number of drug cases worked in their career as a direct measure for the accuracy of a proffered conclusion. They may cite the number of cases worked in their career for the purpose of establishing, defending, or describing their qualifications or experience.
- An analyst shall not use the expressions "reasonable degree of scientific certainty," "reasonable scientific certainty," or similar assertions of reasonable certainty in either reports or testimony

7.9 COMPLAINTS

See *ASCL-DOC-01 Quality Manual*.

7.10 NONCONFORMING WORK

See *ASCL-DOC-01 Quality Manual*. The Chief Forensic Chemist retains records of simple corrections for the section.

7.11 CONTROL OF DATA AND INFORMATION MANAGEMENT

See *ASCL-DOC-01 Quality Manual*.

[5] When hypergeometric distribution sampling is conducted, the analyst can testify to the visual consistency of the sub items within the population. The analyst can testify to the number of randomly selected and independently tested sub-items and their individual results. The analyst must communicate at a confidence level of 95% that 90% of the sub-items statistically contain the reported substance.

8 MANAGEMENT SYSTEM REQUIREMENTS

See *ASCL-DOC-01 Quality Manual*.

9 TEST METHODS

This section describes the testing techniques commonly utilized for the analysis of evidence exhibits in the Forensic Chemistry Section. Strategies for analyzing specific types of evidence exhibits for suspected drug(s) are addressed in the training manual.

If it becomes necessary to make a deviation from a documented method or procedure, it must be technically justified and authorized by the appropriate supervisor. The deviation will be documented in the case record.

If a prosecutor needs to be contacted on how to proceed based on preliminary results, the analyst shall have the data reviewed by another competent analyst before contacting the prosecutor. Documentation of the data review shall be included in the case file.

9.1 TESTING REQUIREMENTS

Minimum testing requirements are listed within this section. Categories of testing are used to address a number of the minimum testing requirements. The categories of common testing techniques are listed in the table below.

Categories for Common Testing Techniques		
Category A	**Category B**	**Category C**
Infrared Spectroscopy (IR), Gas Chromatography/Mass Spectrometry (GC-MS), Energy Dispersive X-Ray Fluorescence (EDXRF)	Gas Chromatography (GC), Thin-Layer Chromatography (TLC), Microscopic Examination	Color Tests, Pharmaceutical Identifiers, pH

If conflicting test results are obtained during the course of testing, this shall be evaluated. Evaluation may trigger re-running of already performed tests, or taking another aliquot of sample to confirm previously obtained test results. If the analyst cannot determine the reason for the conflicting results, a supervisor or the technical leader shall be consulted.

9.1.1 MINIMUM TESTING PER EXHIBIT

For items selected for analysis, at a minimum, two tests per item must be performed in order to report *Test Results* for that item on the *Report of Laboratory Analysis* generated at the conclusion of testing[6]. Minimum testing for identification of compounds and exceptions are addressed below.

[6] i.e., In order to report results other than "not tested," "*element(s)"* name, "acidic solution/basic solution," or "identified as *drug*" on the *Report of Laboratory Analysis*, the minimum testing requirements per exhibit listed in this section must be met.

Newly encountered or unfamiliar substances shall be evaluated to determine if the proper reference material needs to be procured to confirm the presence of the substance.

9.1.1.1 MINIMUM TESTING FOR IDENTIFICATION OF CONTROLLED/PENALTY COMPOUNDS (EXCLUDING ITEMS SUSPECTED TO CONTAIN THC/CBD)

For each controlled compound identified in an item, the analyst shall have, at a minimum, two positive tests for that compound. One of these tests must be from category 'A'. If only two tests are performed, the Category 'A' test shall be a GC-MS with broad temperature program, unless there is substantial reason for choosing a shorter method. (e.g., suspected mushroom material, suspected LSD sugar cubes) The second test may be from Category 'A' or 'B', but not Category 'C'.

The exception to this requirement is tablets/capsules that are pharmaceutically identified and not analytically tested. Reasons for identification only include:

- Tablets were identified to contain a drug that does not substantiate the highest charge
- Tablets were identified to contain a substance that breaks down on the GC-MS to another drug or does not chromatograph well (examples: clorazepate, modafinil, chlordiazepoxide)

During the course of minimum testing, if results indicate that a substance contains more than one controlled substance there is no obligation to exhaustively confirm the presence of all the controlled substances present. At a minimum, the drug that results in the highest penalty level must be confirmed. If more than one drug satisfies the highest penalty level requirement the chemist may look at additional factors (e.g., availability of reference material, suitable test methods, etc.) when selecting which compound to identify.

9.1.1.2 MINIMUM TESTING FOR ITEMS SUSPECTED TO CONTAIN THC OR CBD

CASES CONTAINING 14 GRAMS OR MORE OF PLANT MATERIAL THAT SUBSTANTIATES THE HIGHEST CHARGE OR 0.5 GRAMS OR MORE THAT IS PROBABLE CAUSE OR POTENTIAL THIRD DRUG

POSITIVE MARIHUANA IDENTIFICATION REQUIREMENTS

1. Positive microscopic test for cystolithic hairs
2. GC-MS semi-quant with Δ9-THC response ratio greater than or equal to 1, where a positive Δ9-THC spectrum was obtained

3. One of the following:
    a. Positive thin-layer chromatography for Δ9-THC
    b. Qualitative gas chromatography for Δ9-THC – this must be done if there are interfering compounds identified via GC-MS analysis (The GCRT should be run on a method with broad temperature parameters to ensure other controlled compounds are not present.)
    c. Modified Duquenois-Levine color test (with no other cannabinoids present and a broad temperature program was used on GC-MS)

PLANT MATERIAL UNDER 14 GRAMS, AND OTHER MATERIALS SUSPECTED TO CONTAIN Δ9-THC/CBD (OILS, RESIDUES, FOOD, WAXES, VAPE CARTRIDGES)

Positive tetrahydrocannabinol (Δ9-THC) identification requires a positive result for Δ9-THC from Gas Chromatography-Mass Spectrometry (GC-MS) analysis and a positive from any one of the following tests:

- Thin-layer chromatography (for Δ9-THC) when no interfering compounds have been identified via GC-MS
- Modified Duquenois-Levine (for cannabinoids) (with no other cannabinoids present)
- Qualitative gas chromatography (positive retention time match for Δ9-THC) (The GCRT shall be run on a method with broad temperature parameters to ensure other controlled compounds are not present if the GCMS screen was run on a short method.)

9.1.1.3 MINIMUM TESTING FOR IDENTIFICATION OF ELEMENTS & NON-CONTROLLED COMPOUNDS

For items where only an element or non-controlled compound is identified, the analyst shall have, at a minimum, one positive category 'A' test and a second test from category 'A' or 'B'. If only two tests are performed, the Category 'A' test shall be a GC-MS with broad temperature program, unless there is substantial reason for choosing a shorter method. (e.g., suspected mushroom material, suspected LSD sugar cubes) The exceptions to this requirement are:

1. The analyst may reach a positive conclusion on the presence and identity of elements in an exhibit based on results from either of these testing schemes:
    - X-Ray Fluorescence testing
    - A positive FTIR result and a flame test consistent with that element
2. The analyst may reach a conclusion that inorganic salts (e.g., ammonium nitrate, sodium phosphate, etc.) are present based off of a positive FTIR result.
3. The analyst may reach a conclusion that lithium is present in an exhibit based on the following tests:

- a positive result for lithium hydroxide or lithium carbonate by IR testing
- results of a flame test consistent with lithium

4. The analyst may reach a positive conclusion to the identity of a non-controlled substance based off of a positive pharmaceutical identification.
5. The analyst may reach a conclusion that ammonia is present in an exhibit based on the following tests:
    - a positive result for ammonia by IR testing
    - results of a Nessler's color test consistent with ammonia

9.1.1.4 ADDITIONAL TESTING REQUIREMENTS FOR ISOMERS AND SALT/BASE FORM

If differentiation between diastereomers (e.g., pseudoephedrine and ephedrine) is desired, or salt/base determination is necessary, infrared spectroscopy testing must be conducted. The IR results must be positive to report the diastereomer or salt/base form. FTIR is not required for reporting if a positive pharmaceutical identification of a stereoisomer was obtained.

Federal sentencing specifies different penalties for cocaine base and cocaine hydrochloride. For cases being federally prosecuted, weighable items in which cocaine is detected must be analyzed by infrared spectroscopy, so that if possible, the cocaine form may be determined for reporting.

9.1.1.5 TAMPERING ANALYSIS

The Forensic Chemistry section tests suspected tampering cases for the presence of drugs only.

9.1.1.6 MONEY

If money is discovered in a case, the analyst will take an inventory of the money and record it in their notes. If money is to be tested for the presence of drug residue, the analyst will do a money shake and test the residue acquired from the shake. Money received as evidence shall not be rinsed.

9.2 WEIGHT MEASUREMENT

9.2.1 SCOPE

Determination of the weight of solids/powders/tablets, liquids, plant material, etc. Mass may be determined on one of three balances: analytical, toploading, or bulk. Weight measurements shall be made in grams or kilograms and recorded in the case notes. Weighing shall be carried out on a performance checked balance appropriate for the sample.

The balance weight ranges and uncertainty for a single measurement are listed in the table below. The uncertainty calculations for multiple weighings and the budgets for uncertainty are located in Qualtrax.

Balance	Weighing Range	Uncertainty of Measurement for a Single Weighing
Analytical	0.0500g-100.0000g	0.0020g
Toploading	5.0g – 2000.0g	0.5g
Bulk (LR/LWL)	400g – 32,000g	4g

Weights recorded in case notes are assumed to be net unless otherwise designated. It is the responsibility of the analyst working the case to clearly label gross weights, calculated net weights, or counts by weight in their notes. Items that are normally consumed (edibles, sublingual films, capsules, etc.) are considered a net weight.

9.2.2 REAGENTS/STANDARDS/CONTROLS

- 5g, 100g, 2000g, 10kg reference standard weights

9.2.3 SAMPLE PREPARATION

TESTED ITEMS

All items that will be tested shall have their initial net weight measured unless the item is a residue.

Exclusions to this requirement are:

- Tablets/capsules pharmaceutically identified to contain no controlled substance may be counted only
- Sealed packages (controlled and non-controlled) may be counted only, but if testing of a unit/tablet occurs, it shall have a net weight taken
- Evidence in manufacturing/tampering cases may not be weighed based off of the chemist's training and experience
- Samples that are color tested, with no further analysis, may have a gross weight measured
- A sample that cannot be readily separated from its container may have a gross weight measured. The notes shall indicate the sample could not be removed from the container.
- Syringes do not require a weight measurement be taken
- Vape cartridges do not require a weight measurement be taken
- Gross weight shall be recorded for paper/patches used to administer drugs and not typically consumed

A reserve weight shall be measured after a portion is taken for analysis. Exceptions for the reserve weight are:

- Situations where the sample mass is negligible compared to the mass of the item, the mass of the sample may be satisfactorily substituted

- Samples that are color tested only
- Patches or papers that have been soaked where a portion was not sampled for analysis

NOT TESTED ITEMS

All not tested items shall have a net or gross weight measured unless the item is a residue. Exceptions to this requirement are:

- Items that are excluded from weight requirement, if tested, are also excluded from weight requirement if they are not tested
- Evidence in manufacturing/tampering cases may not be weighed based off of the chemist's training and experience

When multi-unit populations are processed and not all of the units are tested, multiple weighings are required. It is the responsibility of the chemist to ensure all weights are clearly labelled and there is no ambiguity to the relationship between the measurement and the items measured.

COUNT BY WEIGHT

For exhibits containing more than 160 tablets or capsules that are consistent in size/shape/markings across the group the analyst may calculate the total number of tablets or capsules present in the exhibit using the following equation. The count by weight shall be truncated.

$$n_{calc} = \frac{m_{total} \times n_{count}}{m_{count}}$$

Where $\boldsymbol{n_{Calc}}$ = total number of tablets/capsules calculated, $\boldsymbol{m_{Total}}$ = total mass of the tablets/capsules measured, $\boldsymbol{n_{Count}}$ = ≥ 160, the number of tablets/capsules counted, and $\boldsymbol{m_{Count}}$ = the mass of $\boldsymbol{n_{Count}}$.

Example: The chemist receives exhibit E1 which is a small pail filled with several thousand blue tablets. An inspection shows that all the tablets are the same size and inscribed with the same markings. All the tablets together have a weight of 241.8136 grams ($\boldsymbol{m_{Total}}$). 160 tablets ($\boldsymbol{n_{Count}}$) are counted out and are measured to have a weight of 10.2121 grams ($\boldsymbol{m_{Count}}$).

The chemist calculates the total number of tablets ($\boldsymbol{n_{Calc}}$):

$$\boldsymbol{n_{Calc}} = (241.8136 \times 160) \,/\, (10.2121) = 3788.66 \text{ tablets}$$

and truncates the answer to report 3788 tablets **by weight**.

TOTAL NET WEIGHT AND MEASUREMENT UNCERTAINTY

For a single exhibit containing multiple items or consecutive multiple exhibits with identical reported test results and weighed on the same balance, the individual weights as recorded may be summed. The uncertainty for the summed weights depends on the number of measurements taken (n) and the uncertainty associated with each weighing operation (u). The reported uncertainty can

be calculated using the equation below. The resulting uncertainty shall be rounded up not truncated. Pre-calculated uncertainties for ***n*** values up to 50 can be found on the Summary sheet of the uncertainty spreadsheet *DRG-DOC-04 MU Budgets*.

Calculating MU for Multiple Weighings

$$U = \sqrt{n \times u^2}$$

U = total uncertainty, n = number of weighings,

u = uncertainty in a single weighing

CALCULATED NET WEIGHTS AND MEASUREMENT UNCERTAINTY

When multi-item populations are sampled and a conclusion may be inferred about the whole population, the net weight of the entire population will be calculated from measurements on the item and sub-items using the equation below.

$$M_{net} = M_{gross} \times \left[\frac{\sum_{n=1}^{n} m_n}{(M_{gross} - \mu_{gross})} \right]$$

M_{net} = net weight of the entire population (calculated), M_{gross} = gross weight of the entire population, n = number of sub-items tested, m = net weight of each tested sub-item, μ_{gross} = entire gross weight of untested sub-items.

The measurement of uncertainty for the calculated net weight is determined by using spreadsheet *DRG-DOC-03 Hypergeometric Calculated Net Weight and MoU*.

9.2.4 QUALITY ASSURANCE/CONTROL MEASURES

Balances may require daily and monthly performance checks. Checks shall be performed with NIST-traceable certified weights.

Maintenance

- **Daily**: clean and level the balance

Other maintenance is done on an as-needed basis and recorded in the balance log.

Performance verification

Routine Daily Balance Checks	
Daily Checks	**Actions**
Is the balance level?	Level the balance
Is the balance clean?	Clean the balance
Has the balance been performance checked?	Weigh and record verification weight
Was the balance within tolerance?*	If no, perform adjustment before use If yes, Balance ready to use
* Analytical balance – 100g ± 0.0005g, 5g ± 0.0002g, Toploading balance – 100g ± 0.1g, 2,000 ± 0.1g Bulk balance –10,000g ± 2g ,2000g ± 1g	

Each chemist's balance(s) will be subjected to the performance checks in the table above on a daily basis before use. The results of the checks and the serial number (or identifying number) of the calibrated weight used for the checks will be recorded on the appropriate log sheet *DRG-FORM-02, 03, 04, 05*. The performance check tolerances will be at least double the tolerance listed on the calibration certificate, but may be administratively set higher.

If the balance fails performance checks or if it is not in tolerance after it has been adjusted, the balance must be removed from service for repair. If a balance needs more than two adjustments per performance check event, a manager or technical lead shall be consulted. After the balance has been repaired, the balance must be leveled and performance checked before it is returned to service. All repairs, maintenance, and standard weights used must be documented on the appropriate log sheet.

If a balance is moved within a laboratory location, it shall be leveled and performance checked prior to use in casework. If the move is significant, the balance may need to settle for up to 24 hours before the performance check. This shall be recorded on the appropriate log sheet. If a balance is transferred to a separate laboratory location, the balance shall be calibrated prior to being put into service.

- **Reference Standard Intermediate Checks**

Reference standards (stainless steel weights) will be checked on a newly calibrated balance, when practicable, to ensure continued conformance to specified requirements. If the weight is found to be outside of the 'as found' tolerances, listed within the calibration portion of this manual, it shall be taken out of service for calibration or replacement.

9.2.5 INTERPRETATION OF RESULTS

9.2.5.1 PRECAUTIONS/POSSIBLE SOURCES OF ERROR

- Place items to be weighed on center of balance
- Make sure the weighing area is free of air drafts
- Use clean and tared weighing vessel
- Balances needs to be on a sturdy surface
- Wear gloves to protect the integrity of the reference standards

9.2.5.2 POSSIBLE SOURCES OF ERROR INCLUDE BUT ARE NOT LIMITED TO:

- Static interference
- Failure to tare or improper taring of the weighing vessel
- Portion of the weighing vessel not on the balance
- Balance will not stabilize

9.2.6 DOCUMENTATION REQUIREMENTS

The weight shall be recorded in case notes. If the initial weight is calculated in some manner, all weighings shall be documented in the case notes and how the calculation was performed shall be clear.

If the chemist must use a balance other than their personal issue (e.g., another chemist's balance or the bulk balance), it is the responsibility of the chemist using the balance to determine whether the required performance checks have has been performed that day. If the balance has not been checked, the required performance checks must be performed and recorded before the balance may be used in casework. The chemist shall indicate in their notes which weighing(s) were done on a balance they do not ordinarily use, and which balance was used.

9.3 GAS CHROMATOGRAPHY–MASS SPECTROMETRY (GC–MS)

9.3.1 SCOPE

This test method is a two part test used to screen a wide range of substances in which the gas chromatography portion separates components in a mixture, and the mass spectrometry portion detects the components.

Gas Chromatography Parameters

Instrument operation parameters are only one factor in obtaining good separation. A wide variety of parameters may be adjusted by the Forensic Chemist with many combinations of parameters producing acceptable separation. The chemist should rely on their education and training concerning the theoretical and practical aspects of gas chromatography in the selection of instrumental parameters. A separation in the resulting chromatogram should be evaluated by the chemist on the basis of efficiency (the narrowness of the peaks), the peak shapes (i.e., whether they tail or front) and the resolution represented.

Some of the acquisition conditions that the chemist may adjust are parameters such as: injection volumes, injector mode (e.g., split, splitless, etc.), temperature [e.g., of the inlet or oven (initial and final, ramps)], and flow rates. Regardless of the actual instrumental conditions the chemist uses, those conditions must be documented so that the resulting data could be reproduced if necessary.

Mass Spectrometer Parameters

Qualitative data shall always be collected in full scan mode with the high mass scanned exceeding the analyte's molecular weight by at least 10 amu.

9.3.2 REAGENTS, STANDARDS AND CONTROLS

Controls and standards used within this test method are described within their portion of the quality assurance/control portion of this test method.

9.3.3 SAMPLE PREPARATION AND ACQUISITION

The sample may be prepared by a solvent dilution or extraction and should not be acidic or basic or contain any solid material. The blank shall be prepared with the same solvent used to prepare the sample. The blank shall be run before the sample with the same acquisition parameters.

The acquisition parameters shall be chosen based on screen testing, chemist's experience, or tablet identification. A broad temperature ramp is required for samples previously run on a shortened method with no controlled substance results.

GCMS casework blank vial positions are as follows:
Methanol 1-5
Methylene Chloride 26-30
Other 51-55

For automated injections, the sample and blank are each placed in auto sampler vials and capped. An aliquot of the sample may either be automatically or manually injected into the instrument using a syringe.

9.3.4 QUALITY ASSURANCE/CONTROL MEASURES

- **As Needed**: change septum and liner, air and water check (recommended after septum and liner change), rinse and refill solvent vials (daily), rinse waste vials (daily), change filaments, clean source, ballast pump
- **Recommended monthly**: Change solvent/waste vials, clean needle guide, clean inlet cap
- **Recommended yearly**: Change rough pump oil, clean inlet, replace gold seal, replace split vent filter, clean fans
- Other maintenance will be performed when the instrument performance deems maintenance necessary. Maintenance shall be recorded on the GC-MS maintenance Log.

Performance verification

- **Monthly:** autotune

Autotune uses PFTBA (Perfluorotributylamine) masses 69, 219, and 502 to optimize and adjust various parameters for the Mass Selective Detector (MSD). A report is generated (FIG. 1). Autotune must be performed at least once a month. Autotunes shall be indexed into the designated ASCL case number for the instrument and recorded on *DRG-FORM-08*.

Autotunes may be performed more frequently if necessary.

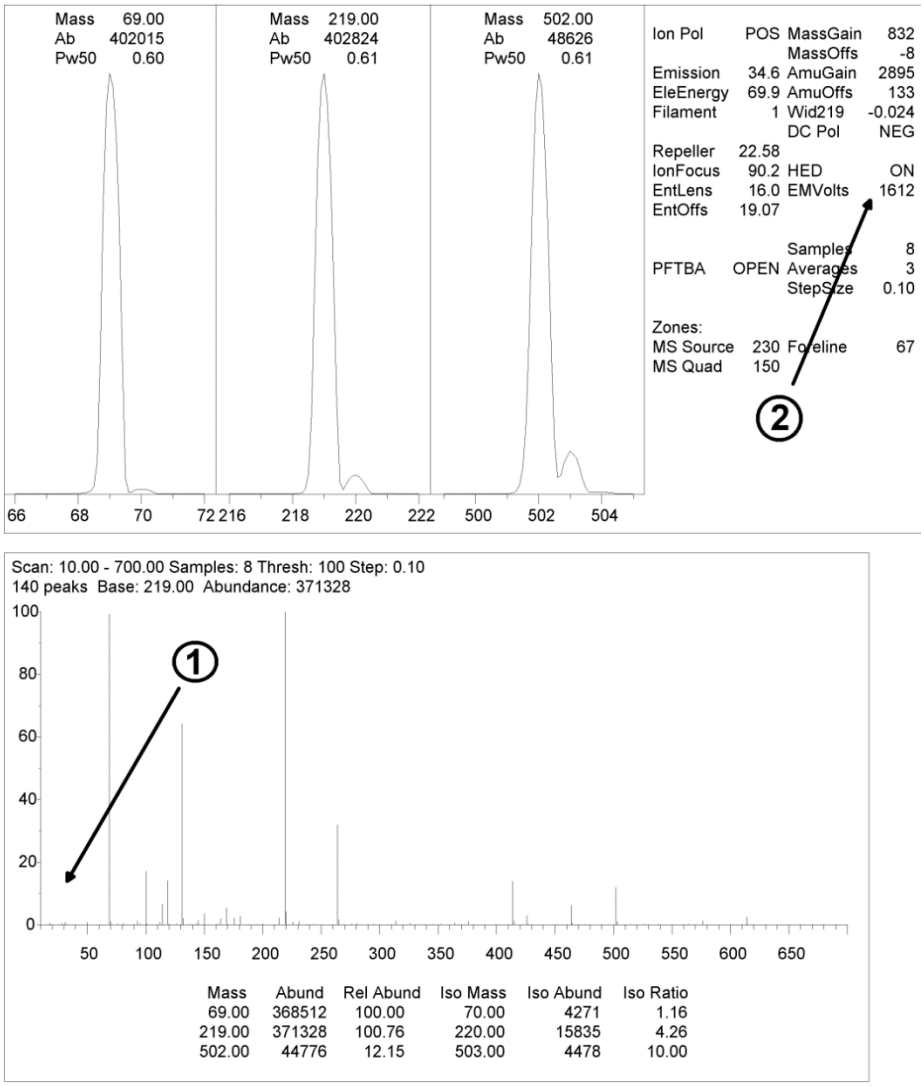


FIG. 1 ***Important Areas of the MSD Report***

The chemist must evaluate the performance check by examining the labeled areas of the report (FIG. 1①,②) for the following conditions:

① Abundance of any peak(s) below 69 m/z (e.g., 18[water], 28[nitrogen], 32[oxygen]) are >20%, relative to the abundance of the peak at mass 69,

② EM voltage > 2500.

If either of these conditions exists, the instrument is not in proper working condition and shall be removed from service until it has been repaired and has passed a performance check.

- **Daily prior to use:** Test Mix
  Test Mix: Analytes in a mixture to include methamphetamine, cocaine, a benzodiazepine, and opiate.

a. Blank shall be run prior to the sample and shall conform to acceptable blank criteria
b. Mixture will be evaluated for acceptable chromatographic peak shapes, abundance of analytes, and positive mass spectrum matches for all analytes
c. Test Mix data shall be indexed into the ASCL case number for the instrument and marked "Pass" or "Fail" on *DRG-FORM-08*.

If substantial changes (absence of an analyte, significant changes in abundance, increased need for subtraction, etc.) occur, notify the appropriate personnel. The instrument should be taken out of service until the issue is resolved and the instrument passes performance checks.

- **Daily for casework sequences**: Sequence Verification
  Another individual shall check the sequence for the following items and initial *DRG-FORM-08.*

  a. Case number and item number
  b. Vial numbers
  c. Methods for blank and sample
  d. Data path and sample name

During the process of loading, the analyst should check the log's previous lines to ensure that the proper documentation for QA/QC has been recorded. If a sequence has been found to be unverified, the analyst shall notify the Chief Forensic Chemist or Technical Leader.

9.3.5 INTERPRETATION OF RESULTS AND REQUIRED DOCUMENTATION

9.3.5.1 PRECAUTIONS TO BE TAKEN

INSTRUMENT:

- Performance check(s)
- Syringe or column blockage

SAMPLE PREPARATION:

- Poor choice of solvent (low analyte solubility) or extraction scheme
- Sample concentration is too dilute
- Insufficient sample taken for analysis
- The sample and reference material should not be acidic or basic or contain any solid material

RUNNING THE SAMPLE:

- Co-eluting compounds
- Proper acquisition parameters

9.3.5.2 POSSIBLE SOURCES OF ERROR

It is the chemist's responsibility to evaluate the chromatogram for any significant peaks. Possible sources of error include:

- Not evaluating all chromatographic peaks
- Co-eluting compounds that create difficulty in identifying substances
- Unsuitable acquisition parameters
- Compounds with similar mass spectrums
- Liner induced compound breakdown (e.g., Δ9-THC to Δ8-THC, 1P-LSD to LSD)
- Extraction induced conversion (e.g., 4-acetoxy DMT to psilocin)

9.3.5.3 CRITERIA FOR POSITIVE, NEGATIVE, AND INDICATIVE RESULTS

The sample shall be evaluated for chromatographic peaks. The evaluation of the total ion chromatogram shall include, but isn't limited to peak shape and peak signal to noise.

Criteria for evaluation of chromatographic peaks:

- Blank: any peak with a signal to noise ≥ 5 should be evaluated
- Sample: any peak with a signal to noise ≥ 10 should be evaluated, exceptions may occur based on analyst's discretion (e.g., weak sample prep, suspected compound that does not chromatograph well)

Once a peak has been determined to need evaluation, the analyst will obtain the mass spectrum and evaluate the fragmentation pattern and fragment ratios prior to comparison to a reference material spectrum.

An acceptable blank will not contain any peaks (signal to noise ≥ 5) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent.

Identification of unknown compound(s) in a sample is based on comparing the sample's mass spectrum to reference spectra. Reference spectra can come from a library, literature, or otherwise-known spectrum. Software matching algorithms are useful for rapidly narrowing the number of possible matches, but ultimate responsibility rests with the chemist to determine whether a sample's mass spectrum matches a given reference spectrum.

Subtractions are permissible provided that the chemist includes the following data in the case file:

- A printout of the original full mass scan for the signal area
- A printout of the full mass scan for the subtraction area
- A printout of the subtraction results

The electronic data files generated from each GC-MS run (samples and blanks) will be retained, at a minimum, until the case is both technically and administratively reviewed.

9.3.5.3.1 POSITIVE RESULTS

The mass spectrum, of a peak in the sample's chromatogram, is visually similar to that of the reference material spectrum and the following criteria are met:

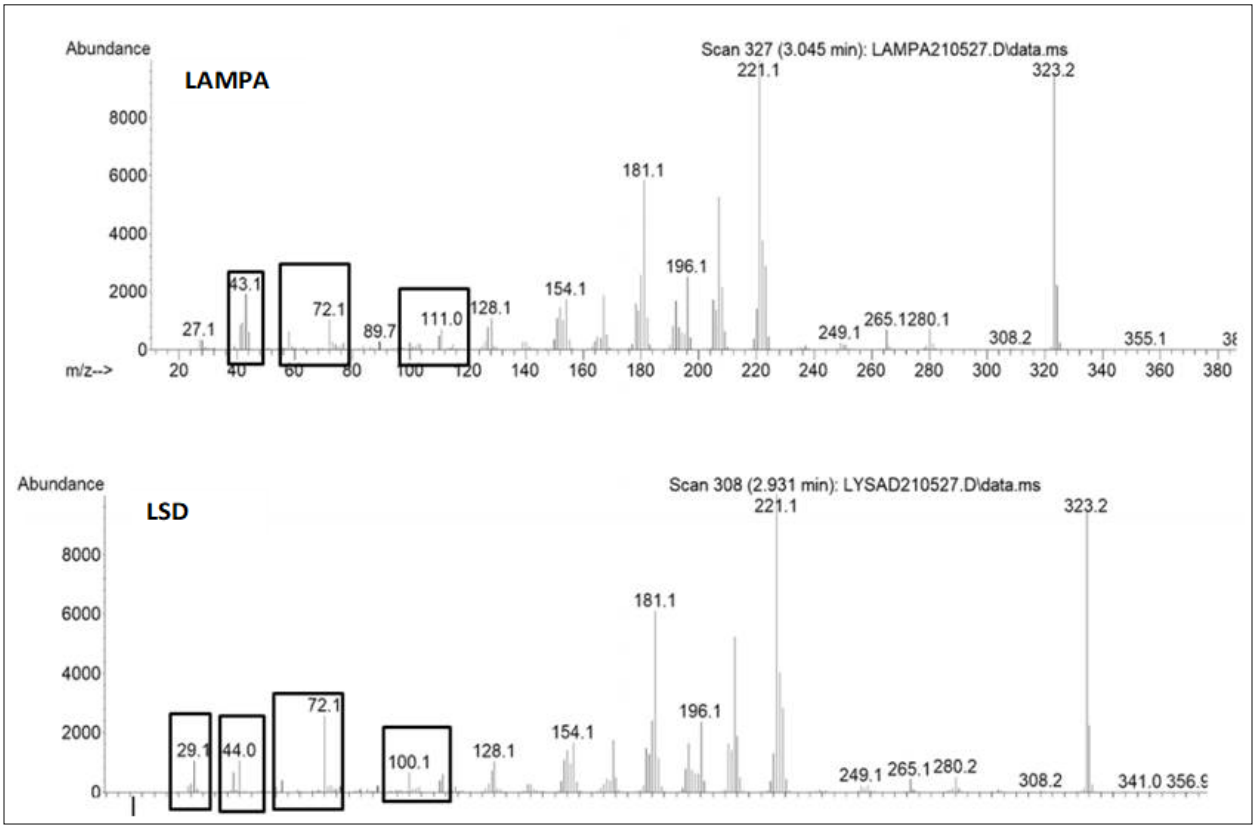
- The signal-to-noise ratio of the chromatographic peak is ≥ 15
- The blank meets acceptable criteria
- If the reference spectrum contains a molecular-ion peak for the compound, the sample's mass spectrum must also contain the molecular ion peak
    - Exception for aliphatic amines – these compounds do not have a strong molecular ion peak and often the M-1 ion is more attainable. For aliphatic amines, the M-1 peak is acceptable for making a positive identification. (aliphatic amine examples – methamphetamine, amphetamine, MDMA, MDA)
    - Exception for compounds where the molecular ion is difficult to obtain : ephedrine/pseudoephedrine, diphenhydramine, fentanyl, and fentanyl analogs
- All peaks present in the reference spectrum shall be present in the sample's spectrum with the following exceptions:
    - Peaks in the reference spectrum that are below the scan limits set in the method parameters [**NOTE:** the appropriate method shall be used for samples suspected to contain compounds such as GBL, GHB, and pregabalin, and LSD which have significant peaks outside the normal scan limits]
    - Peaks in the reference spectrum that are higher in mass than the molecular-ion or the molecular-ion isotopic peaks (if applicable)
    - Low abundance ions (below 10% of the abundance of the base peak) may be absent unless the ion is also the molecular ion
- There shall not be any extra peaks in the sample's spectrum when compared to the reference spectrum with the following exceptions:
    - Low background peaks (below 10% of the abundance of the base peak) are ignored
    - If the reference spectrum has a limited scan range, the sample spectrum shall be compared to a different reference or a reference material spectrum can be acquired for comparison

Other things to consider

- The ion ratios in the sample and reference material are reasonably consistent (this is important in the following scenarios - cannabinoids, cathinones, synthetic cannabinoids, and LSD/LAMPA)

Distinguishing between LSD and LAMPA – see table and mass spectra below
(Samples must be run on LSD method so that it scans low enough to detect 29 fragment)

LAMPA	LSD
29 fragment (likely present but not significant)	29 fragment (significant fragment, should be greater than 10% base peak)
44 fragment **<** 43 fragment	44 fragment **>** 43 fragment
58**:**72 ratio (72 fragment is roughly double)	58**:** 72 ratio (72 fragment is significantly larger)
100 fragment **<** 110/111 fragments	100 fragment **≈** 110/111 fragments



9.3.5.3.2 INDICATIVE RESULTS

The following qualify a sample to be considered indicative:

- The signal-to-noise ratio of the chromatographic peak is ≥10
- The mass spectrum of a peak in the sample's chromatogram is visually similar to that of the reference material spectrum, but the sample's mass spectrum doesn't meet all of the criteria for a positive result

9.3.5.3.3 NEGATIVE RESULTS

The mass spectrum of a peak in the sample's chromatogram does not visually match any available reference material spectra or the sample does not meet the signal to noise requirement to be indicative or positive.

9.3.6 REQUIRED DOCUMENTATION FOR GC-MS RESULTS

9.3.6.1 GENERAL

For exhibits subjected to more than one GC-MS test, the chemist will develop a way to relate the sample preparation/test results in the case notes to the corresponding instrumental printout(s).

Instrumental Printouts
Images of chromatograms and mass spectra (blanks, reference materials, and samples) supporting the analyst's conclusions must be incorporated into the electronic case file (e.g., by scanning or printing to the JusticeTrax Indexer program) before the case request status is marked 'Draft Complete' in LIMS-plus. The unique ASCL case number, exhibit number, vial number, and date must be visible on the image. For runs utilizing a nonstandard method, a copy of the instrumental parameters (method) must also be incorporated into the electronic case file.

9.3.6.2 POSITIVE RESULTS

Any compound(s) meeting the criteria for positive results may be entered into the case notes by chemical name or an appropriate abbreviation. Controlled substances meeting the criteria for positive results must be entered in the case notes by chemical name or an appropriate abbreviation[7]. If no controlled substances are present, other significant substances must be entered into the case notes by chemical name or appropriate abbreviation.

9.3.6.3 INDICATIVE RESULTS

Any compound(s) meeting the criteria for indicative results may be entered into the case notes by chemical name or an appropriate abbreviation followed by a question mark and the notation will be enclosed in parentheses. Controlled substances meeting the criteria for indicative results must be entered in the case notes by chemical name or an appropriate abbreviation.[8]

9.3.6.4 NEGATIVE RESULTS

If the chromatogram contains no peaks or the mass spectra of all peaks in the sample's chromatogram do not visually match any available reference material spectra the results shall be recorded in the case notes. (e.g., "no peaks", "no significant peaks", "no match", "no ID", "no

[7] Known breakdown products and manufacturing byproducts that are controlled are excluded from this requirement
[8] Ibid

controlled substances detected (NCSD)") If the chemist desires to list the best software algorithm match to the mass spectra of peaks in the sample to available library reference spectra, the match shall be enclosed in brackets.

9.4 GAS CHROMATOGRAPHY–RETENTION TIME MASS SPECTROMETER DETECTOR (GC–RT)

9.4.1 SCOPE

Gas Chromatography is used qualitatively for its ability to measure retention times of analytes which can be compared to retention times of known reference materials.

Instrument Operation Parameters

Instrument operation parameters are only one factor in obtaining a good separation. A wide variety of parameters may be adjusted by the chemist, with many combinations of parameters producing acceptable separation. The chemist should rely on their education and training concerning the theoretical and practical aspects of gas chromatography in the selection of instrumental parameters. A separation in the resulting chromatogram should be evaluated on the basis of efficiency (the narrowness of the peaks), the peak shapes (i.e., whether they tail or front) and the resolution represented.

Some of the acquisition conditions that the chemist may adjust are parameters such as: injection volumes, injector mode (e.g., split, splitless, etc.), temperature [e.g., of the inlet or oven (initial & final, ramps)], and flow rates. Regardless of the actual instrumental conditions the chemist uses, those conditions must be documented so that the resulting data could be reproduced if necessary.

9.4.2 REAGENTS, STANDARDS, AND CONTROLS

Controls and standards used within this test method are described within their portion of the quality assurance/control portion of this test method.

9.4.3 SAMPLE PREPARATION AND ACQUISITION

The sample(s) and any necessary reference material(s) should be prepared at approximately the same concentration. The sample and reference material should not be acidic or basic or contain any solid material. Blanks shall be prepared with the same solvent(s) used to prepare the sample and reference material respectively. The blank shall be run before the sample with the same acquisition parameters.

The reference material used for comparison can be run before or after the sample, as long as it has the same acquisition parameters.

If multiple reference materials are mixed in a vial, the mass spectrum for the comparison reference material, used for its retention time, shall be incorporated into the case record.

Reference materials must be run the same date of sample +\- 1 day.

9.4.4 QUALITY ASSURANCE/CONTROL MEASURES

Because this test is performed on GCMS instrumentation, the quality assurance/control measures are the same as those listed in 9.3.4.

9.4.5 INTERPRETATION OF RESULTS

9.4.5.1 PRECAUTIONS TO BE TAKEN

INSTRUMENT:

- Performance check(s)
- Syringe or column blockage

SAMPLE PREPARATION:

- Poor choice of solvent (low analyte solubility) or extraction scheme
- Sample concentration is too dilute
- Insufficient sample taken for analysis
- The sample and reference material should not be acidic or basic or contain any solid material

RUNNING THE SAMPLE:

- Co-eluting compounds
- Proper acquisition parameters

9.4.5.2 POSSIBLE SOURCES OF ERROR

- Co-eluting compounds
- Proper acquisition parameters
- Sample and reference materials should be approximately same concentration

9.4.5.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

GENERAL

The chromatograms of the blanks for samples and reference materials must not contain any peaks (signal to noise ≥5) that are a positive retention time match to the reference material or sample's

analyte of interest. The analyte peak of the chromatograms for samples and reference materials must have a signal-to-noise ratio ≥ 15.

The qualitative analysis of an unknown substance by GC is accomplished by matching the retention time of an unknown sample to the retention time of a known reference material using the following calculation: $\%_{RTT} = \left|\frac{t_{sample} - t_{reference\ material}}{t_{reference\ material}}\right| \times 100$

The acceptable tolerances are listed in the table below (Table 9.3.5.3).

TABLE 9.3.5.3 Maximum Retention Time Match Tolerances	
Retention Time	**Tolerance**
≤ 3 minutes	2% relative
> 3 minutes	1% relative

9.4.5.3.1 CRITERIA FOR POSITIVE RESULTS

The calculated retention time tolerance of the sample versus reference material is ≤ the acceptable tolerance listed in TABLE 9.3.5.3 and the analyte peak of the chromatograms for sample and reference material has a signal-to-noise ratio ≥15.

9.4.5.3.2 CRITERIA FOR NEGATIVE RESULTS

The calculated retention time tolerance of the sample versus reference material is > than the acceptable tolerance listed in TABLE 9.3.5.3 and/or the analyte peak of the chromatogram for sample and/or reference material has a signal-to-noise ratio < 15.

9.4.6 REQUIRED DOCUMENTATION FOR GC QUALITATIVE RESULTS

9.4.6.1 GENERAL

For exhibits subjected to more than one GC test, the chemist will develop a way to relate the sample preparation/test results in the case notes to the corresponding instrumental printout(s).

The retention time tolerance calculation(s) and reference material designation(s) shall be incorporated into case file. Calculations must be done on *DRG-FORM-30 RT Calculations*. If the retention time of the sample and reference are the exact same number, no calculation worksheet is necessary.

Instrumentation:

Images of chromatograms (blanks, reference materials & samples) supporting the analyst's conclusions must be incorporated into the electronic case file (e.g., by scanning or printing to the JusticeTrax Indexer program) before the case request status is marked 'Draft Complete' in

JusticeTrax LIMS-plus. The unique ASCL case number, exhibit number, date, and vial number must be visible on the image (The exhibit number is not required on reference material blanks and reference materials). For runs utilizing a nonstandard method, a copy of the instrumental parameters (method) must also be incorporated into the electronic case file. (Only one copy per method per case file is necessary and must be treated as examination records).

9.4.6.2 POSITIVE RESULTS (GC QUALITATIVE)

Positive test results shall be recorded in the case notes in a manner similar to "Positive (+) retention time (t_R) match for *compound or* by listing the *compound*."

9.4.6.3 NEGATIVE RESULTS (GC QUALITATIVE)

Negative test results shall be recorded in the case notes in a manner similar to "Negative (-) retention time (t_R) match for *compound*".

9.5 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

9.5.1 SCOPE

The methods in this document describe various techniques used to prepare samples and obtain infrared spectra, precautions, and possible sources of error, data interpretation, and notations specific to this test. This test is intended to be supportive of other testing in most instances.

9.5.2 REAGENTS, STANDARDS, AND CONTROLS

Controls and standards used within this test method are described within their portion of the quality assurance/control portion of this test method.

9.5.3 SAMPLE PREPARATION AND ACQUISITION

ACQUISITION PARAMETERS

Routine Instrument Parameters for FTIR*	
Number of Scans	8
Resolution	4.000 cm^{-1}
Sample Gain	Auto
Scanning Range	4000-400 cm^{-1}
* These shall be considered starting point values only and may be adjusted by the chemist depending on the type of information needed.	

9.5.3.1 ATTENUATED TOTAL REFLECTANCE (ATR) EXPERIMENTS

PREPARATION

Normally no sample preparation is needed to acquire infrared spectra of samples in ATR experiments. If an extraction is necessary to remove specific interfering substances, this will be documented in the case notes.

ACQUISITION

A blank spectrum shall be acquired before every sample, by screwing the anvil down onto the diamond crystal and acquiring a spectrum.

Solid Samples
Solids are applied directly to the diamond crystal. The anvil is screwed down into position forcing the sample against the crystal. The spectrum is acquired.

Liquid Samples
Liquids are applied directly to the diamond crystal. Since liquids fully coat the crystal no pressure from the anvil is required. Volatile liquids may be covered with the supplied cover to prevent evaporation. The spectrum is acquired.

9.5.3.2 TRANSMISSION EXPERIMENTS – VAPOR PHASE TECHNIQUE

ACQUISITION & PREPARATION

A blank spectrum shall be acquired by placing a clean vapor phase cell in the sample chamber before each vapor phase IR sample (a vapor phase cell can be cleaned by wiping the cell with a clean wiping paper and heating the cell).

After the blank is acquired, the sample may be prepared in one of the following ways:

- A piece of wiping paper or a piece of filter paper is placed in the cell (in a manner that will not impede the IR beam) and a few drops of the sample solvent are placed on the paper.
- The vapor cell is held over the volatile liquid for a few seconds and the cell is closed.

Once the sample is prepared, the cell is placed in the sample chamber and the spectrum is acquired.

9.5.4 QUALITY ASSURANCE/CONTROL MEASURES

The maintenance and performance verification requirements are listed below for all FTIR instruments.

Maintenance

Maintenance will be performed when the instrument performance deems maintenance necessary and shall be recorded on *DRG-FORM-06.*

Performance verification

The performance verification of each FTIR must be checked each month the instrument is used and after any maintenance has been performed. There are many ways to verify that the instrument is functioning properly depending on the instrument model, instrument location (Little Rock or Lowell), and software version. The FTIR bench and ATR accessory are separate accessories and may require independent verification depending on need. If only the FTIR bench is in use, then verification of the bench is all that is needed. If the ATR accessory is in use, both the bench and ATR accessory must pass verification. Treat all ATR accessories gently when removing or inserting them into the FTIR.

Regardless of the instrument, the results of the performance checks shall be indexed into the instrument case record and recorded on *DRG-FORM-06.* If the instrument fails the performance check, additional checks may be performed. The instrument shall be removed from service if a passing result cannot be obtained. This verification shall be performed prior to returning an instrument to service.

Nicolet iS20 FTIR with ATR Accessory:

1. Remove the ATR accessory.
2. Align the bench (>Collect> Experiment Setup>Diagnostic>Align).
3. Run the Valpro Qualification (>Analyze>Valpro Qualification> Nicolet iS20 KBr – Factory (CP, JP, PHEUR, PV, USP).
4. A ValPro Qualification Report will be generated. If all tests have passed, index the qualification report to the current year's electronic case record for the instrument. Document the results of tests on the logsheet.
5. Replace the ATR accessory on the instrument.
6. Run the Valpro Qualification (Analyze>Valpro Qualification> Smart iTX accessory – PHEUR). When prompted, place the "lolipop" polystyrene standard on the diamond crystal and use the anvil to tighten down into position.
7. A ValPro Qualification Report will be generated. Index the qualification report to the current year's electronic case record for the instrument. Document the results of test on the logsheet.
8. If the instrument failed any of these tests, the instrument must be removed from service for repair.

Nicolet iS10 FTIR with ATR Accessory – Little Rock:

1. Remove the ATR accessory.
2. Place the transmission plate in the FTIR (a screen should appear indicating 'transmission experiment setup')
3. Align the bench (>Collect> Experiment Setup>Diagnostic>Align).
4. Run the Valpro Qualification (>Analyze>Valpro Qualification> Nicolet iS10 KBr – EP).
5. A ValPro Qualification Report will be generated. If all tests have passed, index the qualification report to the current year's electronic case record for the instrument. Document the results of tests on the logsheet.
6. Remove the transmission plate and replace the ATR accessory on the instrument.

7. Run the Valpro Qualification (Analyze>Valpro Qualification> Smart Diamond Accessory-EP). When prompted, place the "lolipop" polystyrene standard on the diamond crystal and use the anvil to tighten down into position.
8. A ValPro Qualification Report will be generated. Index the qualification report to the current year's electronic case record for the instrument. Document the results of test on the logsheet.
9. If the instrument failed any of these tests, the instrument must be removed from service for repair.

9.5.5 INTERPRETATION OF RESULTS

9.5.5.1 PRECAUTIONS TO BE TAKEN

INSTRUMENT

- Verify that all necessary performance verifications have been done and that the instrument has passed each one
- Poor bench alignment which is characterized by:
  - For a background spectrum, the %T at 4000 cm^{-1} approaches zero
  - and/or after a sample spectrum has been baseline corrected, the baseline still "rolls" (i.e., the sample peaks appear on top of a decaying sinusoidal wave)

SAMPLE PREPARATION

- Sample(s) are prepared in too dilute a form (**IDEAL**: The strongest peak will have an absorbance of at least 0.6 or %T of 25.)
- Sample(s) are prepared in too concentrated a form (**IDEAL**: The strongest peak will have an absorbance of no more than 1.2 or %T of 6.)
- Samples containing interfering compounds may require an extraction or other clean-up procedure to remove the interference

RUNNING THE SAMPLE(S)

- Unusual matches suggested by the software matching algorithm: Check which search libraries are selected
- The spectrum contains incompletely subtracted background peaks (e.g., H_2O absorptions at 3800 and 1600 cm^{-1}, and CO_2 absorptions at 2350 and 668 cm^{-1}): Collect a new background and re-run sample

9.5.5.2 SOURCES OF ERROR

- Interfering compound
- Insufficient subtraction of interfering compound
- Not enough sample for acquisition

9.5.5.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

GENERAL

Identification of an unknown sample is based on comparing the sample's infrared spectrum with reference spectra. Care shall be taken to evaluate the sample spectrum for peaks of interest prior to comparing to the reference spectrum. Software matching algorithms are useful for rapidly narrowing the number of possible matches, but ultimate responsibility rests with the Forensic Chemist to determine whether a sample's infrared spectrum matches a given reference spectrum.

If a sample contains multiple infrared active compounds, extraction(s) or other clean-up techniques (or an entirely different testing technique) may need to be employed in order to positively identify these compounds.

Subtractions are permissible provided that the chemist includes the following data in the case file:

- A printout of the original full sample spectrum with or without library match
- A printout of the full subtracted spectrum
- A printout of the subtraction results

An acceptable blank will not be a positive match for a controlled substance, a drug, or a common cutting agent, or contain any significant peaks.

9.5.5.3.1 POSITIVE RESULTS

The sample's infrared spectrum visually matches that of the reference material spectrum. All peaks present in the reference material spectrum are also present in the sample's spectrum with exception of peaks in the sample that may be masked by interfering compounds.

9.5.5.3.2 NEGATIVE RESULTS

The sample should be called negative if:

- The sample visually matches the reference material spectrum, but one or more reference material peaks are missing in the sample spectrum. (This does not include peaks that could be masked by interfering compounds.)
- The sample doesn't visually match any available reference material spectrum

9.5.6 DOCUMENTATION REQUIREMENTS

9.5.6.1 GENERAL

For exhibits subjected to more than one IR test, the chemist will develop a way to relate the sample preparation/test results in the case notes to the corresponding spectral image in the electronic case file.

CASE NOTES
The Forensic Chemist will include in the case notes or instrumental printout, for each IR test performed, the following information:

- Type of technique (ATR assumed unless otherwise noted)
- Type of sample preparation (Direct assumed unless otherwise noted)
- Acquisition technique employed (e.g., s, l, g) (Solid assumed unless otherwise noted)

INSTRUMENT PRINTOUTS
Images of spectra supporting the analyst's conclusions must be incorporated into the electronic case file (e.g., by scanning or printing to the JusticeTrax Indexer program) before the case request status is marked 'Draft Complete' in LIMS-plus. The unique ASCL case number, exhibit number, and acquisition date must be visible on the image.

9.5.6.2 POSITIVE RESULTS

Any compound(s) or mixture meeting the criteria for positive results will be entered into the case notes by chemical name or an appropriate abbreviation.

9.5.6.3 NEGATIVE RESULTS

Samples meeting the criteria for negative results will be entered into the case notes with a clear designation such as "No match" or "No ID." If the chemist desires to list the best software algorithm match(s) of the sample spectrum to available library reference spectra, the match(s) shall be enclosed in brackets. e.g., [sodium bicarbonate]

9.6 THIN LAYER CHROMATOGRAPHY

9.6.1 SCOPE

Thin layer chromatography is a separation technique used within Forensic Chemistry. The methods in this document describe the selection of a TLC solvent system, various aspects of the technique, precautions and possible sources of error, data interpretation and notations specific to this test.

9.6.2 REAGENTS, STANDARDS, AND CONTROLS

Specific reagents or controls are described elsewhere within this test method.

9.6.2.1 SOLVENT SYSTEMS FOR TLC

A wide variety of solvent systems are described in TLC literature. The following table lists the most common solvent systems used; however the use of any published TLC solvent system is acceptable.

Common TLC Solvent Systems		
System	**Makeup**	**Useful For…**
Davidow	Davidow solution[1]:Ammonium Hydroxide (95:5)	Wide variety of acidic, basic and neutral drugs
T1	Methanol: Ammonium Hydroxide (95:5)	Wide variety of acidic, basic and neutral drugs
Hexane/Ether	Hexanes[2]: Diethyl Ether (80:20)	Cannabinoids
Steroids	Methylene Chloride[3]: Ethyl Acetate (80:20) **OR** Methylene Chloride[3]: Methanol (90:10)	Steroids
[1] Ethyl Acetate: Methanol (85:10). [2] Petroleum Ether or Ligroin may be substituted. [3] Chloroform may be substituted.		

Usually a 100 mL portion of the selected solvent system is prepared and transferred to a labeled glass tank lined with filter paper and fitted with a lid.

9.6.3 SAMPLE PREPARATION AND ACQUISITION

9.6.3.1 SAMPLE PREPARATION

Solid samples are dissolved in an appropriate solvent (samples being subjected to Hexane/Ether thin layer shall be extracted in methanol unless methanol is inappropriate). Liquid samples may be used as is or diluted in an appropriate solvent. Some samples may require an extraction procedure to remove interfering compounds.

9.6.3.2 SAMPLE APPLICATION

- A line is drawn with a pencil parallel to, and at least 2 cm from, the bottom of the TLC plate
- The samples are spotted on this line, called the origin, starting at least 2 cm from the side of the plate and at least 1 cm from each other
- The sample and reference material spots are labeled uniquely. The chemist must be able to correlate the sample spot with the case and exhibit number
- The sample(s) and reference material(s), in an adequate volume, are applied with a capillary tube

9.6.3.3 RUNNING THE PLATES

- The TLC plate is placed in a vertical position in a tank containing the selected solvent system so that the application line (origin) is above the level of the mobile phase
- Normally the plate is allowed to develop through a distance of 10-15 cm
- When the development period is complete, the plate is removed from the tank and allowed to dry before visualization

9.6.3.4 VISUALIZATION FOR TLC

As most organic compounds are colorless, they must be made visible so that their relative retention factors can be compared, preferably by a non-destructive technique. There are a wide variety of visualization techniques available, depending on the compound of interest. Visualization techniques that are generally used are described in the table below.

TLC plates run for the analysis of items suspected to contain THC shall

1. Contain the appropriate cannabinoid reference materials
2. Have their baselines covered during the application of Fast Blue BB spray
3. Have the baseline be sprayed with acidified iodoplatinate.

If the visualization of an item with acidified iodoplatinate indicates (by comparison to the reference material usually on or near the baseline) that another controlled substance may be present, further testing (GC-MS analysis at a minimum) is required.

Common TLC Plate Developing Techniques		
Visualization Technique	**Useful For…**	**Comments**
Ultraviolet (UV) Light	Wide variety of organic molecules	Use before any reagent indicator sprays, circle spots in pencil (**NOTE:** The ability to see a given compound may be pH dependant.)
Fast Blue BB	Cannabinoids	Heat plate after spraying
Ninhydrin	Primary and secondary amines	Heat plate after spraying
Acidified Iodoplatinate	Primary through tertiary amines, quaternary ammonium compounds	Useful for overspraying a plate previously sprayed with Ninhydrin or Fast Blue BB (cool plate before spraying)
PMBA	Ergot alkaloids, tryptamines	Heat plate after spraying (required for samples containing LSD and psilocin/psilocybin)
Ethanol: H_2SO_4 (4:1)	Steroids	Heat plate after spraying

9.6.4 INTERPRETATION OF RESULTS

9.6.4.1 PRECAUTIONS TO BE TAKEN

SAMPLE PREPARATION

- Choose an appropriate solvent for the analyte(s) of interest
- Perform extractions on samples that may contain interfering compounds

SAMPLE APPLICATION

- Spot shall be no more than ~4 mm in diameter or resolution will be lost
- The plate surface should not be cut or gouged by the applicator
- It is essential that the spot be dry at the end of application, especially if the solution contains water. Even a small amount of a polar solvent adsorbed on the plate can drastically alter chromatographic properties.

RUNNING THE PLATES

- Overdeveloping the plates may lead to excessive zone (spot) broadening causing secondary problems such as:
    - Weak samples may "disappear"
    - Concentrated samples may overlap with spots in neighboring lanes
- Under developing the plate will result in poor separation for complex samples
- Use of stale solvent system tanks or the improper selection of solvent system may result in poor chromatography

VISUALIZATION

The maximum amount of data is gained from a TLC plate when multiple visualization techniques are used. Poor planning on the order of visualization techniques may lead to data loss.

9.6.4.2 SOURCES OF ERROR

SAMPLE PREPARATION

- Sample is too dilute resulting in no spot
- Poor choice of solvent for analyte resulting in no spot

SAMPLE APPLICATION

- Failure to spot the appropriate or any reference material on the plate

VISUALIZATION

- Sample contains 2 compounds with similar retention factors and they overlap upon visualization (examples listed below):
    - meth/MDMA/codeine
    - Δ8-THC/Δ9-THC/(6aR,9S) Δ10-THC
    - (6aR,9R) Δ10-THC /Δ6a,10a-THC
    - cocaine/fentanyl/multiple fentanyl analogs/eutylone
- Reference materials do not produce visual spots

9.6.4.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

9.6.4.3.1 GENERAL

Identification of compounds by TLC is accomplished by matching the relative retention factors and visualization reaction(s) of known reference materials run simultaneously and on the same plate as the samples.

If a sample contains compounds with similar relative retention factors and visualization reactions, selection of a different solvent system and/or visualization techniques (or an entirely different testing technique) may need to be employed in order to differentiate these compounds.

9.6.4.3.2 CRITERIA FOR POSITIVE RESULTS

A compound in a sample matches the relative retention factor and visualization reaction(s) of a reference material on the same plate.

9.6.4.3.3 CRITERIA FOR NEGATIVE RESULTS

No spots that match a reference material are visible in the sample lane or no spots are present in the sample lane.

9.6.5 DOCUMENTATION REQUIREMENTS

9.6.5.1 GENERAL

The Forensic Chemist will include in the case notes, for each TLC test performed, the following information:

- Type of solvent system used
- Visualization technique(s) employed (e.g., UV, Ninhydrin, etc.)
- The date of the testing (running, visualizing, and re-visualizing the plate will be considered as being performed on the same date unless otherwise noted)

9.6.5.2 DOCUMENTATION OF POSITIVE RESULTS

Any compound(s) meeting the criteria for positive results will be entered into the case notes by chemical name or an appropriate abbreviation. The designation of the reference material used to yield a positive result shall be recorded.

9.6.5.3 DOCUMENTATION OF NEGATIVE RESULTS

Any sample(s) meeting the criteria for negative results will be entered into the case notes in one of the following ways. Samples containing no spots may be entered as “negative” or “no spots”. If

spots are present, the number of spots shall be recorded in the notes. At least one designation of the reference materials run with the sample shall be recorded.

9.7 PHARMACEUTICAL IDENTIFICATION

9.7.1 SCOPE

The methods described in this document can be used to aid in the identification of pharmaceuticals, in the form of tablets and capsules, submitted for drug analysis. This method is used to presumptively identify commercial pharmaceutical products based off of characteristics/appearances only.

9.7.2 REAGENTS, STANDARDS, AND CONTROLS

There are no reagents, standards, or controls associated with this test.

9.7.3 SAMPLE PREPARATION

There is no sample preparation associated with this test. The physical appearance of the tablet/capsule (e.g., imprint, color, shape, scoring, etc.) is required to do the comparison and is required to be in the case notes.

9.7.4 QUALITY ASSURANCE/CONTROL MEASURES

The Forensic Chemistry section has a set list of reference sources that are allowed for pharmaceutical identification. They are listed below:

- Arkansas State Crime Lab Retained Tablet Library (this is the preferred method of identification if a retained tablet is available)
- DIB (any year)
- IdentaDrug (any year)
- Poison Control
- Drugs.com (pill identifier only)
- Manufacturer sources of information (conversations, e-mails, and manufacturer produced ID books)

9.7.5 INTERPRETATION OF RESULTS

9.7.5.1 PRECAUTIONS TO BE TAKEN/SOURCES OF ERROR

- Pharmaceuticals containing similar imprint information
- Counterfeit items

- Imprints matching multiple identifications

9.7.5.2 CRITERIA FOR POSITIVE, INDICATIVE, AND NEGATIVE RESULTS

9.7.5.2.1 POSITIVE RESULTS

The active ingredient(s) of the tablet/capsule have been identified by matching the physical appearance of the tablet/capsule to a reference source.

9.7.5.2.2 INDICATIVE RESULTS

Broken, partial, or worn tablets or damaged capsules largely match a reference source but some markings are not visible.

9.7.5.2.3 NEGATIVE RESULTS

The active ingredient(s) of the tablet/capsule could not be identified because information from reference sources failed to match the physical appearance of the tablet.

9.7.6 DOCUMENTATION REQUIREMENTS

The imprint as well as color, shape, and/or scoring of the tablet/capsule must be documented in the case notes.

9.7.6.1 POSITIVE RESULTS

The active ingredient(s) of any tablet/capsule identifications meeting the criteria for positive results will be entered into the case notes by name or an appropriate abbreviation. The dosage and reference source(s) used to make the identification, and date of identification will also be documented in the case notes. For online sources, only the identification and date need to be recorded in the case notes. The online identification shall be indexed into the case record; the indexed file shall be identifiable to the compared item(s) and contain the date the identification was performed.

9.7.6.2 INDICATIVE RESULTS

The possible active ingredient(s) of any tablet/capsule identifications meeting the criteria for indicative results will be entered into the case notes by name or an appropriate abbreviation followed by a question mark, and the notation will be enclosed in parentheses. The dosage and reference source(s) used to make the identification, and date of identification will also be documented in the case notes. For online sources, only the identification and date need to be recorded in the case notes. The online identification shall be indexed into the case record; the indexed file shall be identifiable to the compared item(s) and contain the date the identification was performed.

9.7.6.3 NEGATIVE RESULTS

If a comparison is used to rule out an ID, the results, source, and date must be documented in your notes. Notes shall also detail the reasons for reaching the negative conclusion. Results will be entered as "negative" or by putting the drug name in brackets. For online sources, only the result and date need to be recorded in the case notes. The comparison information shall be indexed into the case record; the indexed file shall be identifiable to the compared item(s) and contain the date the comparison was performed.

9.8 COLOR TESTING

9.8.1 SCOPE

The methods in this document describe different color tests commonly used and how to perform those color tests. The results of color tests are indicative of the presence or absence of various drug classes and/or organic functional groups. The particular color test(s) used by the Forensic Chemist are usually indicated by the type of sample. Color tests are normally used to help plan future testing of the sample (e.g., appropriate extraction method, instrumentation parameters). If color tests are performed and no additional testing is done, it must be clearly communicated on the report.

9.8.2 REAGENTS, STANDARDS, AND CONTROLS

There are multiple reagents used for color testing. They are listed within this method and the reference materials used to performance check them are listed within the reagent preparation instructions in Qualtrax. Controls, if used, are listed with the specific color test technique.

9.8.3 SAMPLE PREPARATION AND ACQUISITION

9.8.3.1 GENERAL PROCEDURE

1. Place the appropriate reagent(s) in a well plate depression (or a new test tube)
2. Add a small amount of sample
3. Add any additional reagents necessary (multiple reagent color tests)
4. Examine the reactants for any changes
5. Record your observations in the case notes

9.8.3.2 COMMON COLOR TESTS

Color tests routinely used are listed below along with any specific modifications to the above method. The analyst is not limited to the following list of color tests. Other published and recognized color test(s) are acceptable and may be used as needed.

9.8.3.2.1 SINGLE REAGENT COLOR TESTS

- Marquis
- *p*-dimethylaminobenzaldehyde (PMBA)

9.8.3.2.2 MULTIPLE REAGENT COLOR TESTS

- Cobalt Thiocyanate/Stannous Chloride
  In step ❶ add Cobalt Thiocyanate, add Stannous Chloride in step ❸

9.8.3.2.3 MODIFIED DUQUENOIS–LEVINE

This test is appropriate for testing for cannabinoids. Solvents normally used for the extraction include pet ether, hexanes, ligroin, methanol, etc.

1. Transfer a portion of the extract to a new labeled test tube. Heat the test tube to reduce the solvent volume if necessary
2. Add approximately 1 mL of Duquenois-Levine reagent
3. Add approximately 1 mL of concentrated hydrochloric acid (HCl)
4. Agitate the solution and observe any color change
5. Add approximately 1 mL of methylene chloride to the solution and agitate
6. Observe the color of the bottom layer and record in the case notes
7. Run THC positive control along with samples. Follow steps 1-5. Record THC reference material designation used in notes

9.8.3.2.4 NESSLER'S – TESTING FOR AMMONIA GAS

1. Draw a portion of Nessler's supernatant into pipet
2. Draw a portion of sample vapor into pipet, while making sure not to make contact between the pipet or its contents and the sample
3. Observe the color change in the pipet
4. Record in the case notes

9.8.3.2.5 FLAME TEST

When a metal salt is introduced into the flame of a Bunsen burner, the metallic ion produces characteristic color in the flame.

1. In a safe area, ignite a Bunsen burner
2. Obtain a piece of nichrome wire with a loop in one end
3. To clean the wire first dip the wire loop into 0.1N hydrochloric acid and then into deionized water
4. Heat the wire loop in the Bunsen burner flame until the wire begins to glow. Repeat steps 3 & 4 until no color is observed in the flame
5. Dip the looped end of the wire into a sample. (The sample may be solid or dissolved in a small amount of deionized water. If the sample is to be used in a solid form it may be helpful to dampen the wire loop with dilute hydrochloric acid before dipping it in the sample.)
6. Place the loop at the tip of the inner cone of the flame and observe the color given off and record in the case notes

9.8.3.2.6 PH TEST

This test is used primarily in manufacturing cases to determine whether a liquid is acidic or basic.

Procedure:

1. Test litmus paper with acid/base to ensure the paper is still working as expected
2. Deposit a small amount of the solution to be tested onto the litmus paper
3. Compare the color on the litmus paper with the pH scale provided on the litmus paper packaging
4. Record the results in the case notes

9.8.3.2.7 4-AMINOPHENOL COLOR TEST FOR CANNABIS PLANT MATERIAL

This is a presumptive screening test for plant material that may aid in efficient sampling of plant material cases. A color reaction of pink or blue is observed depending on the concentration of certain cannabinoids. A pink color[9] can indicate cannabidiol (CBD) is in higher concentration than other cannabinoids, while a blue color can indicate Δ9-tetrahydrocannabinol (Δ9-THC) is in higher concentration than other cannabinoids.

Procedure:

1. Add approximately 25 mg of the plant material sample to a test tube
2. Add approximately 1 mL of methanol
3. Vortex for approximately 3 seconds
4. Add 1-3 drops of the extracted material to a new test tube
5. Add approximately 1 mL of 4-AP Solution A and 4 drops of 4-AP Solution B to the test tube
6. Vortex for approximately 3 seconds
7. Document color result after 2 minutes (if the color is not discernibly pink or blue, a result of inconclusive is acceptable)

If only this color test is performed, there is no need to record a reserve weight as we know approximately how much is necessary for the test. It is also assumed that you performed a methanol extraction to do this test, therefore, it is not required to be recorded in the notes unless it will also be used for other testing.

Sources of error specific to this color test include but are not limited to:

- Too much or not enough plant material used for the test can affect the color intensity
- Too much methanol added for extraction can affect the color intensity
- Too much 4-AP Solution A can affect the color intensity

[9] The color result of the test does not necessarily correlate with whether the sample is hemp or marihuana, as some samples were found to have a higher CBD concentration and yielded a pink color, but were determined to be marihuana after full testing during the validation.

- Recording results before sufficient time has elapsed (up to 5 minutes was tested in the validation and found to be consistent with the suggested 2 minutes)

Known limitations:

- Blue color is observed when CBN, Δ8-THC, THCV, or Δ9-THC is the most abundant cannabinoid
- Pink color is observed when CBD or CBG is the most abundant cannabinoid

These limitations should have no bearing on the overall outcome of the testing, since this color test is used as a guide on sampling and further testing.

9.8.4 QUALITY ASSURANCE/CONTROL MEASURES

All reagents are verified prior to use in case work. The Duquenois-Levine test has a positive control run with it at the time of the test. The application of the reagents to the spot well or test tube acts as a negative control or blank.

9.8.5 INTERPRETATION OF RESULTS

9.8.5.1 PRECAUTIONS TO BE TAKEN

- Ensure correct order of addition of reagents, if it is a multiple reagent test
- Ensure sufficient sample is introduced to the spot well for testing
- Make sure reagent is added before sample

9.8.5.2 SOURCES OF ERROR

- Dirty spot well could result in false positive
- Poor choice of color test for sample
- Reagent is close to or beyond its usable timeframe
- Color of sample or liquid may interfere with observed results

9.8.5.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

A color change is considered a positive result. No color change is considered a negative result. For pH test, there is no positive or negative result; the analyst will record the pH as the number on the scale the observed color most closely matches. For the 4-aminophenol color test, a result of inconclusive may also be acceptable if differentiation between pink and blue cannot be made.

9.8.6 DOCUMENTATION REQUIREMENTS

The case notes shall include:

- The test performed

- The date of the test
- The result of the test

9.9 MORPHOLOGICAL MICROSCOPY OF PLANT MATERIAL

9.9.1 SCOPE

This method is employed to examine plant material for the presence of cystolithic hairs.

9.9.2 REAGENTS, STANDARDS, AND CONTROLS

N/A

9.9.3 SAMPLE PREPARATION

Direct microscopic examination is the most routine type of analysis. A solvent rinse may be employed, if the sample is moldy, degraded, burned/covered in ash, or very resinous.

9.9.4 QUALITY ASSURANCE/CONTROL

Microscopes are cleaned and/or serviced as needed.

9.9.5 INTERPRETATION OF THE RESULTS

The presence of the following features may be noted:

- Longitudinally grooved stalks and stems
- The top surface (adaxial) of the leaves are darker than the bottom surface (abaxial)
- Compound, palmate leaves with an odd number of leaflets (typically seven)
- Leaflets are serrated (pointing toward tips) and pointed at both ends
- Ovoid, mottled seeds (typically brown) that have a ridge around the greatest circumference
- Seeds contain a white flesh that resembles coconut flesh
- Glandular trichomes (with or without stalk, bulbous)
- Non-glandular trichomes (non-cystolithic)

9.9.5.1 PRECAUTIONS TO BE TAKEN AND POSSIBLE SOURCES OF ERROR

- Not acquiring a sufficient sample for examination (i.e, size of sample or quality of sample taken)
- Not prepping a sample that needs rinsing
- Dirty microscope oculars
- Inadequate lighting
- Improper focus
- Sample is too finely ground

9.9.5.2 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS AND REQUIRED DOCUMENTATION

9.9.5.2.1 POSITIVE RESULTS

Presence of cystolithic hairs attached to leaf material is a positive result for microscopic examination. The results shall be recorded in the case notes.

9.9.5.2.2 NEGATIVE RESULTS

Absence of cystolithic hairs attached to leaf material is a negative result for microscopic examination. The results shall be recorded in the case notes.

9.10 SEMI-QUANTITATIVE DETERMINATION OF Δ9-THC

9.10.1 SCOPE

This method is for semi-quantitative evaluation of Δ9-tetrahydrocannabinol (Δ9-THC) in plant material, using liquid extraction and agitation followed by analysis on a gas chromatograph-mass spectrometer (GCMS). A qualitative result may be determined by comparison to the response of a decision point standard, using the internal standard tribenzylamine (TBA).

This method should only be employed in the following scenarios where the individual item(s) to be tested contains at least 0.5g of plant material:

- There is at least fourteen grams of plant material present in the case, and the material substantiates the highest charge for that case or defendant (In cases where at least 14 grams of plant material are present with other items, such as edibles or wax, that are of equal charge, the plant material shall be selected as part of the testing.)
- There is at least 0.5 grams of plant material present that is probable cause, a potential manufacturing case, or a potential third drug

9.10.2 REAGENTS, STANDARDS, AND CONTROLS

Certified reference materials shall be used to establish traceability. These will be purchased from a provider that is ISO 17034 accredited.

A 1% decision point shall be prepared and run with each batch of semi-quant casework samples. The decision point and samples must all use the same preparation of internal standard.

Decision Point Preparation

1. Pipet 500 µL of Δ9-THC CRM into an auto-sampler vial using a calibrated micropipette
2. Add 1000 µL of TBA semi-quant internal standard solution using a calibrated micropipette

3. Cap vial and agitate to mix

TBA Semi-Quant Internal Standard Stock Solution Preparation

1. Weigh out approximately 0.125 g of TBA
2. Add to a 1000 mL class A volumetric flask
3. Fill to volume with methanol
4. Agitate until completely dissolved
5. Record preparation on prep form

Note: Different volumes of the TBA stock solution may be prepared as needed.

Extracted Blank Preparation

1. Add 1000 µL of semi-quant TBA internal standard solution to an auto-sampler vial
2. Cap vial

9.10.2.1 SAMPLE PREPARATION

1. Weigh out 95–105 mg of plant material and add it to a test-tube
2. Record the amount in the case record
3. Add 2000uL of the TBA semi-quant internal standard solution using a calibrated micropipette
4. Vortex for approximately one minute and allow to extract for ten minutes
5. Filter extract and transfer approximately 1 mL to an auto-sampler vial and cap

Sample preparation and acquisition may be carried out by another competent analyst to facilitate batching samples for analysis. Transfer of the pre-weighed sample to the analyst performing the extraction shall be noted on the Semi-Quant batch worksheet.

9.10.2.2 ACQUISITION

The data is acquired using the THC method in scan mode using the following ions:

Δ9-THC

- quantitative ion 299
- qualitative ions 231 and 314

TBA

- quantitative ion 210
- qualitative ions 196 and 287

An extracted blank (negative control) will be run with each batch, to evaluate the materials used in the extraction for contamination. This sample comprises only the materials used in the extraction, with no plant material added. An acceptable extracted blank will not contain any peak in the retention time area of Δ9-THC with indicative mass spectrum for Δ9-THC or any other peak (signal

to noise ≥ 5) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent.

A solvent blank shall be run before each case sample to detect carryover. An acceptable blank will not contain any peaks (signal to noise ≥ 5) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent.

A solvent clean shall be run after each extracted case sample to help prevent carryover.

A decision point shall be prepared, using a Δ9-THC CRM and the TBA stock solution, and run before each batch of samples.

The sequence format for a decision point and samples is:

Sequence order	***Sample***	***Purpose***
1	Solvent clean	Clean inlet/column
2	Extracted blank	Evaluate extraction process for contamination
3	Solvent clean	Clean inlet/column
4	Solvent blank	Evaluate for carryover
5	Decision Point	Establish Decision Point
6	Solvent clean	Clean inlet/column Evaluate for carryover Analyze sample
7	Solvent blank	
8	Case 1	
9	Solvent clean	Clean inlet/column Evaluate for carryover Analyze sample
10	Solvent blank	
11	Case 2	

The decision point and extracted samples may be analyzed up to three days after their extraction/initial analysis.

9.10.3 QUALITY ASSURANCE/CONTROL MEASURES

All QA/QC requirements for the GCMS instrument also apply for semi-quantitative testing. (i.e., monthly autotune, daily test mix, daily solvent vial exchange, rinsing of waste vials, sequence verification)

QUARTERLY PIPETTE CHECKS

Pipettes shall be checked quarterly at a minimum. A low and high value shall be checked on the 20-300 µL pipettes. A mid-range and high value shall be checked on the 100-1000 µL pipettes.

Pipette check procedure:

1. Obtain beaker of E-pure water
2. Take temperature of water
3. Record temperature on the appropriate pipette check spreadsheet

4. Enter the Z factor for recorded temperature into the appropriate cell on the spreadsheet (make sure you have it recorded as 1/Z factor)
5. Tare a beaker on the balance and begin to aliquot portions of liquid into the beaker
6. Record the weight on the spreadsheet of each portion for 10 trials
7. Check to make sure the % error is below the acceptable error in section 6.4.7.1 of this manual
8. If the error is too high, the error shall be evaluated and the check may be performed again
    a. If it still fails, the equipment shall be taken out of service until maintenance is performed and performance checks pass
    b. If it passes, perform a 3rd check which must also pass. If it does not, the equipment shall be taken out of service until maintenance is performed and performance checks pass

9.10.4 INTERPRETATION OF RESULTS

9.10.4.1 PRECAUTIONS TO BE TAKEN

- Ensure complete integration of all analyte and internal standard peaks
- Ensure proper labels are entered in sequence table for decision point, blanks, and samples
- Ensure the decision point (calibrator) is set up and you have selected quantitate for the sample

9.10.4.2 POSSIBLE SOURCES OF ERROR

Potential errors in decision point preparation:

- Omission of internal standard
- Improperly pipetting reference material or internal standard
- Reference material concentration (e.g., bad batch/concentration is incorrect)

Potential errors in sample preparation:

- Incomplete transfer of sample to extraction vessel
- Improperly pipetting internal standard
- Omission of internal standard
- Inadequate extraction time/process

9.10.4.3 DATA INTERPRETATION

There must be no reportable analyte peak in the extracted blank (negative control) for the batch results to be acceptable.

An acceptable blank will not contain any peaks (signal to noise ≥ 5) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent.

The responses of TBA and THC shall be compared and shall be between 20 – 50% using the following equation:

$$DP\ response\ ratio = \frac{TBA\ area\ response}{THC\ area\ response}\ x\ 100$$

Semi-quant calculation shall not be run on any samples that do not have a positive mass spectrum for Δ9-THC.

9.10.5 REQUIRED DOCUMENTATION

Each case record shall contain:

- Extracted blank with mass spectral data
- Decision point blank and sample data
- Casework blank and sample data
- Casework semi-quant report(s)
- Semi-quant results
- Batch worksheet with:
  - Preparation date
  - Case number and item number
  - Pipettes used
  - CRM manufacturer and lot number
  - TBA designation
  - Initials and date documenting transfer of sample for analysis, if applicable

9.11 QUANTITATIVE DETERMINATION OF Δ9-THC

9.11.1 SCOPE

The method described here is for quantitative determination of Δ9-tetrahydrocannabinol in plant material via gas chromatography-mass spectrometry. The quantitative determination is achieved by a comparison of the response of an internal standard, tribenzylamine (TBA). The range for quantitation is from 0.1% - 1.0% Δ9-THC and the measurement uncertainty is 20 % of the determined value.

9.11.2 REAGENTS, STANDARDS, AND CONTROLS

Certified reference materials shall be used to establish traceability. These will be purchased from a provider that is ISO 17034 accredited.

CALIBRATION CURVE AND POSITIVE CONTROL PREPARATION

A new calibration curve will be prepared each week quantitation will be performed.

Calibrator Preparation Procedure:

1. Pipette specific calibrator volume of Δ9-THC into an auto-sampler vial with a calibrated micropipette
2. Add 25 µL of TBA with a calibrated micropipette
3. Add methanol to equal a total volume of 1mL
4. Cap vial and agitate to mix

Level	% Δ9-THC	TBA (µL)	Δ9-THC (µL)	Methanol (µL)
1	0.1	25	50	925
2	0.2	25	100	875
3	0.3	25	150	825
4	0.6	25	300	675
5	1.0	25	500	475
Positive Control	0.4	25	200	775

Positive Control Preparation Procedure:

1. Pipet 200 µL (0.4% THC) of Δ9-THC into an auto-sampler vial with a calibrated micropipette (should be separate lot and preferably different manufacturer than calibrator source)
2. Add 25 µL of TBA with a calibrated micropipette
3. Add 775 µL methanol
4. Cap vial and agitate to mix

Extracted Blank Preparation Procedure:

1. Add 25 µL of TBA to an auto-sampler vial
2. Add 975 µL of methanol
3. Cap vial and agitate to mix

INTERNAL STANDARD PREPARATION

5.0 mg/mL TBA preparation procedure:

1. Weigh out approximately 1.0 g of TBA
2. Add to 200 mL class A volumetric flask
3. Fill to volume with methanol
4. Agitate until dissolved
5. Record preparation on prep form

Note: Different volumes of the TBA stock solution may be prepared as needed.

9.11.3 SAMPLE PREPARATION AND ACQUISITION

9.11.3.1 SAMPLE PREPARATION

PLANT MATERIAL

1. Take an adequate representative sample by collecting multiple small portions from throughout the plant material to ensure the sample is greater than 1 gram
2. Dry the representative sample in an oven with the temperature set to 50°C, for a minimum of four hours, until completely dry
3. Homogenize approximately 1 gram of the sample using a small blender for at least one minute
4. Weigh out approximately 100 mg of homogenized plant material and transfer to a screw-top vial
5. Record the amount in the case record
6. Add 50 µL of TBA to the screw-top vial with a calibrated micropipette
7. Add 1.95 mL of methanol to the screw-top vial
8. Vortex for approximately 1 minute and then sonicate for 15 minutes
9. Filter extract with cotton and transfer approximately 1 mL into an auto-sampler vial and cap

NOTES:

1. Each item to be quantitated will have two separate tubes prepared for quantitation.
2. A calibrated pipette and proper technique shall be used for all liquid measurements.
3. Weighed amount may be adjusted based on evaluation of the Δ9-THC load on previous GCMS analysis to ensure the response of the sample falls on the curve when quantitated.

9.11.3.2 ACQUISITION

The data is acquired using the THCQuant method with selective ion monitoring (SIM) mode using the following ions:

Δ9-THC

- quantitative ion 299
- qualitative ions 231 and 314

TBA

- quantitative ion 210
- qualitative ions 196 and 287

A solvent blank shall be run before each case sample to detect carryover. There must be no peak in this blank with a response greater than 2% of the compound for which it is the blank.

A “solvent clean” will be run after each extracted case sample to help prevent carryover. This data need not be evaluated, but shall be verified during the sequence verification process.

The sequence format for a calibration curve and samples is therefore:

Sequence order	*Sample*	*Purpose*
1	Solvent clean	Clean inlet/column
2	Extracted blank	Evaluate extraction process for contamination
3	Solvent clean	Clean inlet/column
4	Solvent blank	Evaluate for carryover
5	Calibrator 1	Establish calibration curve
6	Solvent blank	Evaluate for carryover
7	Calibrator 2	
8	Solvent blank	
9	Calibrator 3	
10	Solvent blank	
11	Calibrator 4	
12	Solvent blank	
13	Calibrator 5	
14	Solvent clean	Clean inlet/column
15	Solvent blank	Evaluate for carryover
16	Positive control - front	Evaluate accuracy of calibration curve. Made from second source.
17	Solvent clean	Clean inlet/column
18	Solvent blank	Evaluate for carryover
19	Case 1, first replicate	Analyze sample
20	Solvent clean	Clean inlet/column
21	Solvent blank	Evaluate for carryover
22	Case 1, second replicate	Analyze sample
23	Positive control - back	Evaluate accuracy of calibration curve. Made from second source.

The sequence format for a subsequent batch of case samples is:

Sequence order	*Sample*	*Purpose*
1	Solvent clean	Clean inlet/column
2	Extracted blank	Evaluate extraction process for contamination
3	Solvent clean	Clean inlet/column
4	Solvent blank	Evaluate for carryover
5	Positive control	Evaluate accuracy of calibration curve. Made from second source.
6	Solvent clean	Clean inlet/column
7	Solvent blank	Evaluate for carryover
8	Case 1, first replicate	Analyze sample
9	Solvent clean	Clean inlet/column
10	Solvent blank	Evaluate for carryover
11	Case 1, second replicate	Analyze sample
12	Positive control - back	Evaluate accuracy of calibration curve. Made from second source.

9.11.4 QUALITY ASSURANCE/CONTROL MEASURES

All QA/QC requirements for the GCMS instrument also apply for quantitative testing. (i.e., monthly autotune, daily test mix, daily solvent vial exchange, rinsing of waste vials, sequence verification)

An extracted blank (negative control) will be run with each batch, to evaluate the materials used in the extraction for contamination. This sample comprises only the materials used in the extraction, with no plant material added. There must be no Δ9-THC peak in this negative control with a response greater than 2% of the lowest calibrator's Δ9-THC response for the batch results to be acceptable.

A positive control targeted at 0.40 % Δ9-THC (w/w) will be run bracketing each batch, to evaluate the calibration curve for accuracy. The results for both positive controls must be within 20% of the targeted value for the batch results to be acceptable. This control must be made from a second source or second lot number, when practicable.

A solvent blank will be run before each case sample to detect carryover. There must be no peak in this control with a response greater than 2% of the compound for which it is the blank.

A "solvent clean" will be run after each extracted case sample to help prevent carryover. This data need not be evaluated, but shall be verified during the sequence verification process.

QUARTERLY PIPETTE CHECKS

Pipettes must be checked quarterly; instructions are located in the Δ9-THC semi-quantitative test method of this manual.

9.11.4.1 CALIBRATION CURVE EVALUATION

The calibration curve shall be set to fit a quadratic line and have a correlation coefficient (r^2) of at least 0.990. Curve points are weighted at 1/x.

Should the curve with all five points not meet the 0.990 correlation coefficient, removal of a curve point may be allowed. Removal of curve points shall be discussed with a supervisor or technical lead prior to proceeding. If a point is removed and there is still not a 0.990 correlation coefficient, the calibrators may be re-run to attempt to achieve a 0.990 correlation coefficient or the curve shall be re-prepared.

The expected ion ratios (*Quant*:*Qual1* and *Quant*:*Qual2*) are determined by averaging the ratios of the five calibrators. The expected ion ratios shall be entered into the data analysis method on Mass Hunter (or equivalent). For an analytical result to be acceptable, all ion ratios shall fall within 20% (relative) of their expected value. If the ion ratio falls outside of this range, further evaluation will be performed to determine whether the results are acceptable[10].

The retention time of the fourth calibrator shall be entered into the Mass Hunter THCQuant method (or equivalent).

The maximum error of the positive control shall not exceed 20% of the targeted value.
Should the error exceed 20%, the cause will be evaluated. The positive control vial may be re-run and if it still exceeds the 20% error, two additional positive controls shall be prepared and analyzed.

Extracted samples may be analyzed up to three days after their extraction/initial analysis.

[10] For example, if the peak has been misintegrated or has a coeluting peak.

9.11.5 INTERPRETATION OF RESULTS

9.11.5.1 PRECAUTIONS TO BE TAKEN

- Ensure the response of each sample falls between the response of the lowest calibrator and the highest calibrator
- Ensure complete integration of all analyte and internal standard peaks
- Ensure the peak ratios of each ion are within 20% of the average

9.11.5.2 POSSIBLE SOURCES OF ERROR

Potential errors in calibration curve and positive control preparation:

- Omission of internal standard
- Improperly pipetting reference material or internal standard
- Reference material concentration (e.g., bad batch/concentration is incorrect)

Potential errors in sample preparation

- Failing to record the weight of the sample aliquot to be quantitated
- Incomplete transfer of sample to extraction vessel
- Improperly pipetting internal standard
- Omission of internal standard
- Not pipetting enough or too much methanol upon bringing to volume

9.11.5.3 EVALUATION OF DATA AND CALCULATIONS

There must be no peak in this control with a response greater than 2% of the compound for which it is the blank. Duplicate samples shall be within 10% of the mean. If the duplicate samples do not meet this requirement, two more aliquots shall be prepped for quantitation.

The following equation is set within the THC Quant Calculation Worksheet:

$$Percent\ difference\ from\ mean\ = \frac{\left([Sample\ 1] - \left(\frac{[Sample\ 1] + [Sample\ 2]}{2}\right)\right)}{\left(\frac{[Sample\ 1] + [Sample\ 2]}{2}\right)} \times 100$$

This worksheet also:

- Calculates the % THC present in the sample with the following equation:

$$\%\ \Delta 9\text{ - }THC = \frac{100 \times Instrumental\ result}{Milligrams\ of\ sample\ used}$$

- Calculates the average of the two aliquots and truncates the result to two significant figures for reporting

- Sums the two aliquot weight determinations for reporting
- Calculates the measurement uncertainty and rounds it up to two significant figures for reporting

9.11.6 REQUIRED DOCUMENTATION

Each case record shall contain:

- Notes page
- Calibration curve with associated correlation coefficient (r^2)
- Mass Hunter batch data table containing appropriate curve, check, and sample(s)
  - Print out of the averaged ions used for THC and TBA for the quant method
  - Print out of the retention time set for THC and TBA for the quant method
- Quant Calculation Worksheet
- Extracted blank, curve, positive control, casework blank and sample total ion chromatograms
- Compound information for qualifier and quantitative ions for each sample, curve point, and positive control (this shows the individual integration of each ion)

9.12 EXTRACTIONS FOR DIFFICULT SAMPLES

9.12.1 REQUIRED EXTRACTIONS

Illicit Tablets (suspected Ecstasy tablets)

1. Sample approximately half of the tablet and crush it
2. Split into two test tubes
3. Extract your GCMS sample in enough solvent to fill a GCMS vial (base extraction is preferable and the analyst may use hexanes as solvent to remove excess caffeine)
4. Filter into GCMS vial

Suspected NBOH Compounds Derivatization

1. Dissolve the sample in Acetonitrile that has been dried over Sodium Sulfate.
2. Add dried Acetonitrile to an empty test tube (for your blank).
3. Remove the liquid from the sample extract and place in a labelled test tube. Add Sodium Sulfate to further dry your sample.
4. Add ~160 µL of sample to a GCMS vial with sleeve. Do the same for the blank.
5. Add ~ 160 µL of BSTFA (1% TMCS) to a GCMS vial sleeve. Do the same for the blank.
6. Cap and mix well
7. Run on broad method
8. If you get poor results, repeat the process trying to reduce air/moisture.

Items containing both 4-acetoxy DMT and psilocin together

It is known that 4-acetoxy DMT hydrolyzes to psilocin with some extraction types. If 4-acetoxy DMT and psilocin are found together in the same GCMS data, the steps, listed below, shall be followed.

1. Extract a new sample using acetonitrile
2. Run on GCMS and assess (if psilocin persists consult a supervisor or technical leader).

9.12.2 SUGGESTED EXTRACTIONS FOR DIFFICULT SAMPLES

Clonazepam tablets

1. Soak in saturated sodium bicarbonate (1-2 hours)
2. Extract with minimal methylene chloride

LSD on sweet tarts or sugar cubes

1. Soak in 0.1N HCl overnight
2. Make basic with saturated sodium bicarbonate
3. Extract into methylene chloride
4. Dry down in a sleeve

Mushroom Extraction

1. Soak in 0.1 N HCl
2. Wash with methylene chloride
3. Take the 0.1 N HCl layer and add saturated sodium bicarbonate to make basic (ph ~ 9 or 10)
4. Add methylene chloride
5. Dry methylene chloride layer down to a sleeve

Mushroom/Chocolate Extraction

1. Sample is transferred into a mortar and ground with a pestle.
2. The resulting powder is covered with 10 % acetic acid (or 0.1N HCl), and the sample is further ground with a pestle.
3. An additional 5 to 7 ml of distilled or E-Pure water is added, and the mixture is ground for about 2 minutes, creating a thin slurry.
4. This slurry is divided into equal portions, and is transferred into large screw top tubes.
5. Approx. 3 ml of CH_2Cl_2 is added to each tube, and the tubes are centrifuged for 3 minutes.
6. The aqueous layer is pipetted into a beaker from each of the tubes.
7. The aqueous solution in the beaker is neutralized by slowly adding solid sodium bicarbonate until the effervescence stops.
8. The pH is checked with pH paper to make sure it doesn't surpass 9.
9. The resulting solution is then transferred into large screw top tubes, and each extracted with approx. 3 ml of CH_2Cl_2.
10. The tubes are centrifuged for about 5 minutes.
11. The CH_2Cl_2 layers are combined in a small beaker.
12. The CH_2Cl_2 extract is concentrated under air (no heat), transferred to a sleeve, and analyzed on the GC/MS.

THC Candy

1. Dissolve sample in hot distilled or E-Pure water
2. Extract in Petroleum Ether.
3. Dry down.
4. Reconstitute in methanol.

Items Suspected to Have GHB (derivatization)

1. Dry ethyl acetate (in a covered Erlenmeyer flask) over sodium sulfate. Dry the solvent at minimum overnight.
2. Dry the sample with air **(NO HEAT or low heat if necessary)**. This step may not be necessary; direct samples have been used yielding positive results.
3. Into three large test tubes (the ones in Tox that have caps) place: 10 mg of GHB standard in one tube; sample in the second tube; the third tube is left empty (this will be used as a negative control). Flush the tubes with nitrogen and cap.
4. Add 1 ml of dried ethyl acetate to each tube. Dry while flushing with nitrogen then immediately cap.
5. Add 100 µl of BSTFA **(record lot #)** to each tube. Flush with nitrogen then immediately cap. Vortex.
6. Incubate at 70º C for approximately 10 minutes.
7. Dilute the samples with dried ethyl acetate and filter with sodium sulfate.
8. Consider using ethyl acetate in GCMS wash vials; methanol can interact with the derivatizing agent.
9. If you get poor results, repeat the process trying to reduce air/moisture.